

Section 3.3

METHOD 4--DETERMINATION OF MOISTURE IN STACK GASES

OUTLINE

<u>Section</u>	<u>Documentation</u>	<u>Number of Pages</u>
SUMMARY	3.3	2
METHOD HIGHLIGHTS	3.3	8
METHOD DESCRIPTION		
1. PROCUREMENT OF APPARATUS AND SUPPLIES	3.3.1	9
2. CALIBRATION OF APPARATUS	3.3.2	19
3. PRESAMPLING OPERATIONS	3.3.3	7
4. ON-SITE MEASUREMENTS	3.3.4	10
5. POSTSAMPLING OPERATIONS	3.3.5	4
6. CALCULATIONS	3.3.6	8
7. MAINTENANCE	3.3.7	3
8. AUDITING PROCEDURE	3.3.8	4
9. RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3.3.9	1
10. REFERENCE METHOD	3.3.10	5
11. REFERENCES	3.3.11	1
12. DATA FORMS	3.3.12	14

397

SUMMARY

A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

This Reference Method is used for the accurate determination of moisture content (as needed to calculate emission data) of stack gas. The Reference Method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the Reference Method or its equivalent. Alternative methods capable of yielding results within 1% water of the Reference Method may be used, subject to the approval of the administrator.

Note: The Reference Method may yield questionable results when applied to saturated gas streams or to gas streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second method for determining the moisture content shall be used simultaneously with the Reference Method, as follows. Assume that the gas stream is saturated. Attach a temperature sensor capable of measuring to $\pm 1^{\circ}\text{C}$ (2°F) to the Reference Method probe. Measure the stack gas temperature at each traverse point during the Reference Method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage either by using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart or by using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods, subject to the approval of the administrator, shall be used.

The procedure described in Method 5 for determining moisture content is acceptable as a Reference Method.

(348)

Section No. 3.3
Revision No. 0
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Page 3 of 11

The Method Description which follows is based on the method promulgated in the Federal Register, Vol. 42, No. 160, August 18, 1977.

A complete copy of the Reference Method is contained in Section 3.3.10. References 1 and 2 in Section 3.3.11 were used in the subsections concerning the description, calibration, and maintenance of the sampling train. Data forms are provided in Section 3.3.12 for the convenience of the Handbook user.

METHOD HIGHLIGHTS

Method 4 is a gaseous sampling method for the determination of water vapor content of stack gas. This method requires fewer quality control activities than the other methods in this Handbook. Since moisture is collected as a gas, the analysis is not easily biased; furthermore, water vapor is not a regulated pollutant. However, an accurate determination of moisture content is usually needed to set and determine the isokinetic sampling rate and also to perform emission data calculations. The accuracy and precision³ of the method have been demonstrated to be acceptable except when applied to saturated gas streams or to streams that contain water droplets.

The blank data forms at the end of this section may be removed from the Handbook and used as checklists during the pretest, field sampling, and posttest operations. Each form has a subtitle (e.g., Method 4, Figure 2.5) to aid the user in locating a similar filled-in form in the Method Description. Items/parameters that can cause significant error are designated with an asterisk on each form.

1. Procurement of Equipment - Section 3.3.1 (Procurement of Apparatus and Supplies) gives the specifications, criteria, and design features for equipment and materials required for performing Method 4 tests. The sampling apparatus has the same design criteria as Method 5 with the exception that a pitot tube system and sample nozzle are not required for collecting the sample. This section is designed as a guide in the procurement and initial check of equipment and supplies. The activity matrix (Table 1.1) at the end of Section 3.3.1 can be used as a quick reference, and follows the same order as the written descriptions in the main text.

2. Pretest Preparations - Section 3.3.2 (Calibration of Apparatus) provides a step-by-step description of the required calibration procedures. The calibration of the Method 4 equipment is similar to that of Method 5 with the exception that

Method 4 sampling is performed at a constant rate not in excess of $0.021 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{min}$). The calibration section can be removed and compiled, along with calibration sections for all other methods, into a separate quality assurance reference manual for use by calibration personnel. A pretest checklist (Figure 2.5) or similar form should be used to summarize the calibration data.

Section 3.3.3 (Presampling Operations) provides the tester with a guide for supplies and equipment preparation for field tests. Sample impingers may be charged in the base laboratory as long as the water-filled impinger section and silica gel impinger are each tightly capped. The pretest preparation form (Figure 3.1) can be used as an equipment checkout and packing list. An important item in the pretest preparation is the determination of stack gas saturation or water droplet content. Under these conditions, a specially calibrated stack gas temperature sensor is required for moisture determination. The methods for packing and the descriptions of packing containers should help protect the equipment, but are not required.

3. On-Site Measurements - Section 3.3.4 (On-Site Measurements) contains a step-by-step procedure for performing sampling and sample recovery. Testing is performed at a constant rate not to exceed $0.02 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{min}$). When the stack gas is suspected of being saturated or having water droplets, the additional procedure for accurately measuring the stack temperature to determine the moisture content with the saturated vapor pressure and absolute stack temperature must be performed and compared with the Reference Method. The on-site measurement checklist (Figure 4.4) is provided to assist the tester with a quick method for checking requirements.

4. Posttest Operations - Section 3.3.5 (Postsampling Operations) gives the posttest equipment check procedures. Figure 5.1 or a similar form should be used to provide a summary of the posttest calibration checks, and should be included in the emission test report. No control samples are required for

analysis since the analysis is only a gravimetric or volumetric determination of a sample which is large enough to provide an easy determination.

Section 3.3.6 (Calculations) provides the tester with the required equations, nomenclature, and suggested number of significant digits. It is suggested that a programmable calculator be used if available to reduce the chance of calculation error.

Section 3.3.7 (Maintenance) provides the tester with a guide for a routine maintenance program. This program is not required, but if performed, should reduce malfunctions.

5. Auditing Procedure - Section 3.3.8 (Auditing Procedure) provides a description of necessary activities for conducting performance and system audits. A performance audit of the data processing and a systems audit of the on-site measurements should provide independent assessments of the quality of data needed to allow the collaborative test results to be used in the final data evaluation.

PRETEST SAMPLING CHECKS
(Method 4, Figure 2.5)

Date _____ Calibrated by _____

Meter box number _____ $\Delta H@$ _____

Dry Gas Meter*

Pretest calibration factor _____ (within 2% of the average factor for each calibration run)

Impinger Thermometer

Was a pretest temperature correction used? _____ yes _____ no
If yes, temperature correction _____ (within 2°C (4°F) of reference value)

Dry Gas Meter Thermometer

Was a pretest temperature correction made? _____ yes _____ no
If yes, temperature correction _____ (within 6°C (10.8°F) of reference value)

Barometer

Was the pretest field barometer reading correct? _____ yes _____ no

Stack Gas Temperature Sensor (if required)*

Was a temperature sensor required for moisture determination purposes? _____ yes _____ no

Was a pretest temperature correction used? _____ yes _____ no
If yes, temperature correction _____ (within $\pm 1^\circ\text{C}$ (2°F) over the entire range)

Did the temperature sensor agree with the reference thermometer (within $\pm 1^\circ\text{C}$ (2°F) over the range of 10° to 82°C (50° to 180°F))? _____ yes _____ no

*Most significant items/parameters to be checked.

(453)

PRETEST PREPARATION CHECKLIST
(Method 4, Figure 3.1)

Apparatus check	Acceptable		Quantity required	Ready		Packed and loaded	
	Yes	No		Yes	No	Yes	No
<u>Probe type</u>							
Borosilicate glass _____							
Quartz glass _____							
Other _____							
Heater and leak checked* _____							
<u>Filter</u>							
In-stack _____							
Out-stack _____							
Glass wool _____							
Other _____							
<u>Condenser</u>							
Impingers _____							
Other _____							
<u>Cooling System</u>							
Ice bath _____							
Other _____							
<u>Metering System</u>							
Vacuum gauge _____							
Checked* _____							
Pump _____							
Leak checked* _____							
Thermometers _____							
Calibrated* _____							
Dry gas meter _____							
Calibrated* _____							
Other _____							

*Most significant items/parameters to be checked.

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6/10/81

Figure 3.1 (continued)

Apparatus check	Acceptable		Quantity required	Ready		Packed and loaded	
	Yes	No		Yes	No	Yes	No
<u>Barometer</u>							
Mercury _____							
Aneroid _____							
Other _____							
Calibrated* _____							
<u>Quantitative Instrument</u>							
Graduated cylinder _____							
Trip balance _____							
Calibrated* _____							
<u>Stack Temperature Sensor*</u>							
Type _____							
Calibrated _____							

*Most significant items/parameters to be checked.

ON-SITE MEASUREMENT CHECKLIST
(Method 4, Figure 4.1)

Procedure used: Reference _____ Approximate _____

Reference Method

Conducted simultaneously with pollutant emission test? _____

Impingers properly placed?* _____

Impinger content: 1st _____ 2nd _____ 3rd _____

4th _____ Modifications _____

Cooling System: Crushed ice _____ Other _____

Sampling time per point _____

Probe heater (if applicable) on? _____ Temp _____

Crushed ice in ice bath? _____

Leak check? (optional) _____ Leakage rate _____

Sampling rate constant (within 10%)*? _____

All data properly recorded?* _____

Posttest leak check?* (mandatory) _____

Leakage rate* _____

Analysis - Impinger Content

Method: Volumetric _____ Gravimetric _____

Measurement of volume of water condensed:

Graduated cylinder _____ Other _____

Measurement of silica gel: Balance _____ Other _____

Color of silica gel? _____ Condition _____

All analytical data properly recorded? _____

*Most significant items/parameters to be checked.

POSTTEST EQUIPMENT CHECKS
(Method 4, Figure 5.1)

Dry Gas Meter

Pretest calibration factor Y _____ (must be within $\pm 2\%$)*
Posttest checks, Y_1 _____ Y_2 _____ (must be within $\pm 5\%$ of
pretest)
Recalibration required? _____ yes _____ no
If yes, recalibration factor Y _____ (must be within $\pm 2\%$)*
Lower calibration factor Y _____ for calculations (pretest
or posttest)*

Dry Gas Thermometer

Was a pretest meter temperature correction used? _____ yes _____ no
If yes, temperature correction _____ (within $\pm 3^\circ\text{C}$ (5.4°F)
over range)*
Posttest comparison with mercury-in-glass thermometer _____
_____ (within $\pm 6^\circ\text{C}$ (10.8°F) at room temperature)
Recalibration required? _____ yes _____ no
Recalibration temperature correction, if used _____ (within
 $\pm 3^\circ\text{C}$ (5.4°F) over range)*
If yes, no correction is necessary for calculations when meter
thermometer temperature is higher
If recalibration temperature is higher, add correction to aver-
age meter temperature for calculations

Barometer

Was pretest field barometer reading correct? _____ yes _____ no
Posttest comparison _____ mm (in.) Hg [within ± 2.5 mm
(0.1 in.) Hg of mercury-in-glass barometer reading]
Was recalibration required? _____ yes _____ no
If yes, no correction is necessary for calculations when the
field barometer has the lower reading
If the mercury-in-glass reading is lower, then subtract the
difference from the field data readings for the calculation

Stack Gas Temperature Sensor (if required)

Posttest comparison _____ [within $\pm 2^\circ\text{C}$ (4°F) of reference
values]*
Was recalibration required? _____ yes _____ no

*Most significant items/parameters to be checked.

(10)

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

A schematic of the sampling train used in Method 4 is shown in Figure 1.1. Commercial models of this train are available. For those who desire to build their own, construction details are published in APTD-0581.¹ Allowable modifications are described in the following sections.

The operating, maintenance, and calibrating procedures for the sampling train are in APTD-0576.² Since correct usage is important to obtaining valid results, all users should read the document and adopt the procedures unless alternatives are outlined herein.

Applicable specifications, criteria, and/or design features are in this section to aid in the selection of equipment which assures collection of data of good quality. Procedures and limits (where applicable) for acceptance checks are given. The descriptive title, the identification number (if applicable), and the results of the acceptance check are recorded in the procurement log, which is dated and signed by the individual performing the check. An example of a procurement log is shown in Figure 1.2, and a blank copy of the log is in Section 3.3.12 for the convenience of the Handbook user. If calibration is required as part of the acceptance check, the data are to be recorded in a calibration log. Table 1.1 at the end of this section is a summary of the quality assurance activities for the procurement and acceptance of apparatus and supplies.

1.1 Sampling Apparatus

1.1.1 Probe - The sampling probe should be a borosilicate (Pyrex), quartz glass, or stainless steel tubing with an outside diameter (OD) of about 16 mm (0.625 in.), and it should be encased in a stainless steel sheath with an OD of 25.4 mm (1 in.). Alternatively, other metals or plastic tubing may be used if approved by the administrator.

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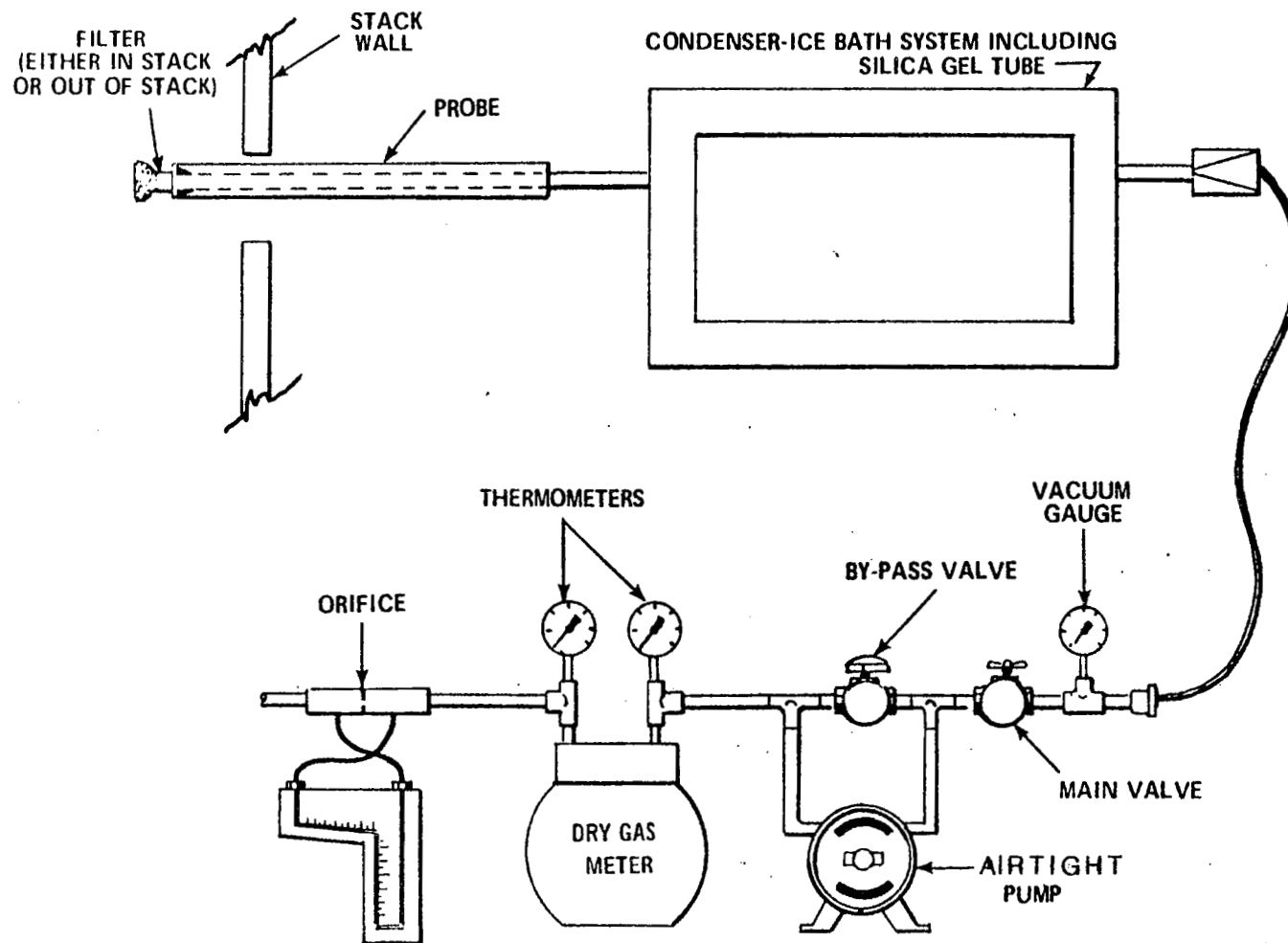


Figure 1.1 Moisture sampling train (Reference Method).

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
Meter Box	2	4687-G	Acme Testing Equipment	6/2/79	8/5/79	\$2,500/ea	Calibrated + Ready	Calibrated By WGD 8/10/79

Figure 1.2 Example of a procurement log.

Either borosilicate or quartz glass liners may be used for stack temperatures up to about 480°C (900°F), but quartz glass liners should be used from 480° to 900°C (900° to 1650°F). Either type of liner may be used at the higher temperatures for short periods of time with administrator approval. However, the absolute upper limits--the softening temperatures of 820°C (1508°F) and 1500°C (2732°F)--for borosilicate and quartz respectively must be observed.

A heating system is required which will maintain an exit gas temperature of 120° ±14°C (248° ±25°F) during sampling. Other temperatures may be specified by a subpart of the regulations and must be approved by the administrator for a particular application. Since the actual probe outlet temperature is not usually monitored during the sampling, probes constructed in accordance to APTD-0581¹ and utilizing the calibration procedures in APTD-0576² will be acceptable.

Upon receiving a new probe, the user should visually check it for specifications: that is, is it the length and composition ordered? The probe should be visually checked for breaks or cracks, and it should be checked for leaks on a sampling train (Figure 1.1). The probe heating system should be checked as follows:

1. Connect the probe with a nozzle attached to the inlet of the pump.

2. Electrically connect and turn on the probe heater for 2 or 3 min. The probe should become warm to the touch.

3. Start the pump and adjust the needle valve until a flow rate of about 0.02 m³/min (0.75 ft³/min) is achieved.

4. Be sure the probe remains warm to the touch. The heater should be capable of maintaining the exit air temperature at a minimum of 100°C (212°F) under these conditions. If it cannot, the probe should be repaired, returned to the supplier, or rejected.

1.1.2 Condenser - Four impingers should be connected in series with leak-free ground-glass fittings or any similarly leak-free

noncontaminating fittings. The first, third, and fourth impingers must be the Greenburg-Smith design modified by replacing the inserts with an unstricted 13 mm (0.5 in.) ID glass tube extending to within 13 mm (0.5 in.) of the flask bottom. The second impinger must be a Greenburg-Smith with the standard tip and plate. Modifications--for example, using flexible connections between impingers, using materials other than glass, or using a flexible vacuum hose to connect the filter holder to the condenser--may be used if approved by the administrator. The fourth impinger outlet connection must allow insertion of a thermometer capable of measuring $\pm 1^{\circ}\text{C}$ (2°F) of true value in the range of 0° to 25°C (32° to 77°F).

Alternatively, any system that cools the gas stream and allows measurement of the condensed water and the water vapor leaving the condenser, each to within 1 ml or 1 g, may be used with approval of the administrator.

Upon receipt of a standard Greenburg-Smith impinger, the user should fill the inner tube with water. If the water does not drain through the orifice in ≤ 6 to 8 s, the impinger tip should be replaced or enlarged to prevent an excessive pressure drop in the sampling system. Each impinger should be checked visually for damage--breaks, or cracks, or manufacturing flaws such as poorly shaped connections.

1.1.3 Temperature Gauge - A thermometer capable of measuring within 1°C (2°F) is located at the outlet of the fourth impinger. The thermometer should be checked upon receipt for damage--for example, dents, bent stem, broken face.

1.1.4 Cooling System - An ice bath container and crushed ice (or equivalent) are needed for condensing the moisture.

1.1.5 Metering System - The metering system should consist of a vacuum gauge; a leak-free vacuum pump; thermometers capable of measuring $\pm 3^{\circ}\text{C}$ (5.4°F) of true value in the range of 0° to 90°C (32° to 194°F); a dry gas meter with 2% accuracy at the required sampling rate; and related equipment as shown in Figure 1.1. Other metering systems capable of maintaining sampling rates

(6/1)

within 10% of constant rate and capable of determining sample volumes to within 2% may be used if approved by the administrator. Sampling trains with metering systems designed for sampling rates higher than that described in APTD-0581¹ and APTD-0576² may be used if the above specifications can be met.

Upon receipt or after construction of the equipment, the user should perform both positive and negative pressure leak checks before beginning the system calibration procedure described in Section 3.3.2. Any leakage requires repair or replacement of the malfunctioning item.

1.1.6 Differential Pressure Gauge - The differential pressure gauge should be an inclined manometer or the equivalent to measure the orifice pressure differential.

Initially, check the gauge against a gauge-oil manometer at a minimum of three points: 0.64 mm (0.025 in.); 12.7 mm (0.5 in.); and 25.4 mm (1.0 in.) H₂O. The gauge should agree within 5% of the gauge-oil manometer. Repair or return to the supplier any gauge which does not meet these requirements.

1.1.7 Barometer - A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg is required.

A preliminary check of a new barometer should be made against a mercury-in-glass barometer or the equivalent. In lieu of this, the absolute barometric pressure may be obtained from a nearby weather service station and adjusted for the elevation difference between the station and the sampling point. Either subtract 2.5 mm Hg/30 m (0.1 in. Hg/100 ft) for an elevation increase or add the same for an elevation decrease from the station value. If the barometer cannot be adjusted to agree within 2.5 mm (0.1 in.) Hg of the reference barometric pressure, it should be returned to the manufacturer.

1.1.8 Graduated Cylinder and/or Triple Beam Balance - A graduated cylinder or triple beam balance may be used to measure the water condensed in the impingers during sampling. Additionally,

the graduated cylinder may be used to measure the water initially placed in the first and second impingers. In either case, the required accuracy is 1 ml or 1 g; therefore, the cylinder must have subdivisions ≤ 2 ml, and the triple beam balance is usually capable of weighing to the nearest 0.5 g.

1.1.9 Stack Gas Temperature Sensor - A thermocouple, thermometer, or equivalent, for measuring the stack gas temperature within $\pm 1^\circ\text{C}$ (2°F) is required when the gas stream is suspected of being saturated or containing water droplets. This accuracy should be in the range of about 10° to 82°C (50° to 180°F). Upon receipt check the specifications and calibrate as described in Section 3.3.2.

6/1/8

Table 1.1 ACTIVITY MATRIX FOR PROCUREMENT AND
ACCEPTANCE OF EQUIPMENT

Apparatus and supplies	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Sampling probe liner	Specified material of construction; equipped with heating system capable of maintaining $120^{\circ} \pm 14^{\circ}\text{C}$ ($248^{\circ} \pm 25^{\circ}\text{F}$)	Visually check, and run heating system	Repair, return to supplier, or reject
Differential pressure gauge (manometer)	Meet criteria (Method 2, Sec 3.1.2); agree within 5% of gauge-oil manometer	Check against gauge-oil manometer at a minimum of 3 points: 0.64(0.025); 12.7 (0.5); 25.4(1.0) mm (in.) H_2O	Repair or return to supplier
Vacuum gauge	Range 0-760 mm (0-30 in.) ± 2.5 mm (0.1 in.) Hg at 380 mm (15 in.) Hg	Check against a mercury U-tube manometer upon receipt	Adjust or return to supplier
Vacuum pump	Leak free and capable of maintaining a flow rate of 0.02-0.03 m^3/min (0.66-1.0 ft^3/min) for pump inlet vacuum of 380 mm (15 in.) Hg	Check upon receipt for leaks and capacity	Repair or return to supplier
Orifice meter	ΔH_Q of 46.74 ± 6.35 mm (1.84 ± 0.25 in.) H_2O ; not mandatory	Upon receipt, visually check for damage, and calibrate against wet test meter	Repair if possible; otherwise return to supplier
Impingers	Standard stock glass; pressure drop across impingers not excessive (Subsec 1.1.6)	Visually check upon receipt; check pressure drop (Subsec 1.1.6)	Return to supplier

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(2/16)

Table 1.1 (continued)

Apparatus and supplies	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Dry gas meter	Capable of measuring total volume within $\pm 2\%$ at a flow rate of $0.02 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{min}$)	Check for damage upon receipt; calibrate against wet test meter (Sec 3.3.2)	Reject if damaged, behaves erratically, or cannot be properly adjusted
Thermometers	Should read within $\pm 1^\circ\text{C}$ of true value in the range of 0°C to 25°C for impinger thermometer, and $\pm 3^\circ\text{C}$ of true value in the range of 0° to 90°C for dry gas meter thermometers	Check upon receipt for dents or bent stem; calibrate against mercury-in-glass thermometer (Sec 3.3.2)	Reject if unable to calibrate
Barometer	Capable of measuring atmospheric pressure within $\pm 2.5 \text{ mm}$ (0.1 in.) Hg	Check against a mercury-in-glass barometer or equivalent; calibrate (Sec 3.1.2)	Determine correction factor or reject if difference is more than $\pm 2.5 \text{ mm}$ (0.1 in.) Hg
Graduated cylinder	Glass, Class-A, 250 ml , subdivisions $\leq 2 \text{ ml}$	Upon receipt, check stock number, cracks, breaks, and manufacturer flaws	Replace or return to supplier
Trip balance	500-g capacity; capable of measuring within $\pm 0.5 \text{ g}$	Check with standard weights upon receipt	As above
Stack gas temperature sensor	Within $\pm 1^\circ\text{C}$ (2°F) in range of 10° to 82°C (50° to 180°F)	Upon receipt check specifications; then calibrate (Sec 3.3.2)	As above


(11)

2.0 CALIBRATION OF APPARATUS

Calibration of the apparatus is one of the most important functions in maintaining data quality. The detailed calibration procedures included in this section are designed for the equipment specified by Method 4 and described in the previous section. A laboratory log book of all calibrations must be maintained. Table 2.1 at the end of this section summarizes the quality assurance activities for calibration.

2.1 Metering System

2.1.1 Wet Test Meter - Wet test meters are calibrated by the manufacturer to an accuracy of $\pm 0.5\%$. The calibration of the wet test meter must be checked initially upon receipt and yearly thereafter. A wet test meter with a capacity of $3.4 \text{ m}^3/\text{h}$ ($120 \text{ ft}^3/\text{h}$) will be necessary to calibrate the dry gas meter. For large wet test meters ($>3\ell/\text{rev}$), there is no convenient method to check the calibration. For this reason, several methods are suggested, and other methods may be approved by the administrator. The initial calibration may be checked by any of the following methods:

1. Certification from the manufacturer that the wet test meter is within $\pm 1\%$ of true value at the wet test meter discharge, so that only a leak check of the system is then required.
 2. Calibration by any primary air or liquid displacement method that displaces at least one complete revolution of the wet test meter.
 3. Comparison against a smaller wet test meter that has previously been calibrated against a primary air or liquid displacement method, as described in Section 3.5.2.
 4. Comparison against a dry gas meter that has previously been calibrated against a primary air or liquid displacement method.
- 

The calibration of the test meter should be checked annually. The calibration check can be made by the same method as that of the original calibration, with the exception that the comparison method need not be recalibrated if the calibration check is within $\pm 1\%$ of the true value. When this agreement is not obtained, then the comparison method or wet test meter must be recalibrated against a primary air or liquid displacement method.

2.1.2 Sample Meter System - The sample meter system--consisting of the pump, vacuum gauge, valves, orifice meter, and dry gas meter--is initially calibrated by stringent laboratory methods before it is used in the field. After the initial acceptance, the calibration is rechecked after each field test series. This recheck is designed to provide the tester with a method that can be used more often and with less effort to ensure that the calibration has not changed. When the quick check indicates that the calibration factor has changed, the tester must again use the complete laboratory procedure to obtain the new calibration factor. After recalibration, the metered sample volume must be multiplied by either the initial or the recalibrated calibration factor that yields the lowest gas volume for each test run.

Before initial calibration of the metering system, a leak check should be conducted. The meter system should be leak free. Both positive (pressure) and negative (vacuum) leak checks should be performed. Following is a pressure leak-check procedure that will check the metering system from the quick disconnect inlet to the orifice outlet and will check the orifice-inclined manometer for leaks:

1. Disconnect the orifice meter line from the downstream orifice pressure tap (the one closest to the exhaust of the orifice), and plug this tap.

2. Vent the negative side of the inclined manometer to the atmosphere. If the inclined manometer is equipped with a three-way valve, this step can be performed by merely turning the

4.11

three-way valve that is on the negative side of the orifice-inclined manometer to the vent position.

3. Place a one-hole rubber stopper with a tube through its one hole in the exit of the orifice, and connect a piece of rubber or plastic tubing to the tube, as shown in Figure 2.1.

4. Open the positive side of the orifice-inclined manometer to the "reading" position. If the inclined manometer is equipped with a three-way valve, this will be the line position.

5. Plug the inlet to the vacuum pump. If a quick disconnect with a leak-free check valve is used on the control module, the inlet will not have to be plugged.

6. Open the main valve and the bypass valve.

7. Blow into the tubing connected to the end of the orifice until a pressure of 127 to 178 mm (5 to 7 in.) H₂O has built up in the system.

8. Plug or crimp the tubing to maintain this pressure.

9. Observe the pressure reading for a 1-min period. No noticeable movement in the manometer fluid level should occur. If the meter box has a leak, a bubbling-type leak-check solution may aid in locating the leak(s).

After the metering system is determined to be leak free by the positive leak-check procedure, the vacuum system to and including the pump should be checked by plugging the air inlet to the meter box. If a quick disconnect with a leak-free stopper system is presently on the meter box, then the inlet will not have to be plugged. Turn the pump on, pull a vacuum within 75 mm (3 in.) Hg of absolute zero, and observe the dry gas meter. If the leakage exceeds $1.5 \times 10^{-4} \text{ m}^3/\text{min}$ ($0.005 \text{ ft}^3/\text{min}$), the leak(s) must be found and minimized until the above specifications are satisfied.

Leak checking the meter system before initial calibration is not mandatory, but is recommended.

Note: For metering systems having diaphragm pumps, the normal leak-check procedure described above will not detect

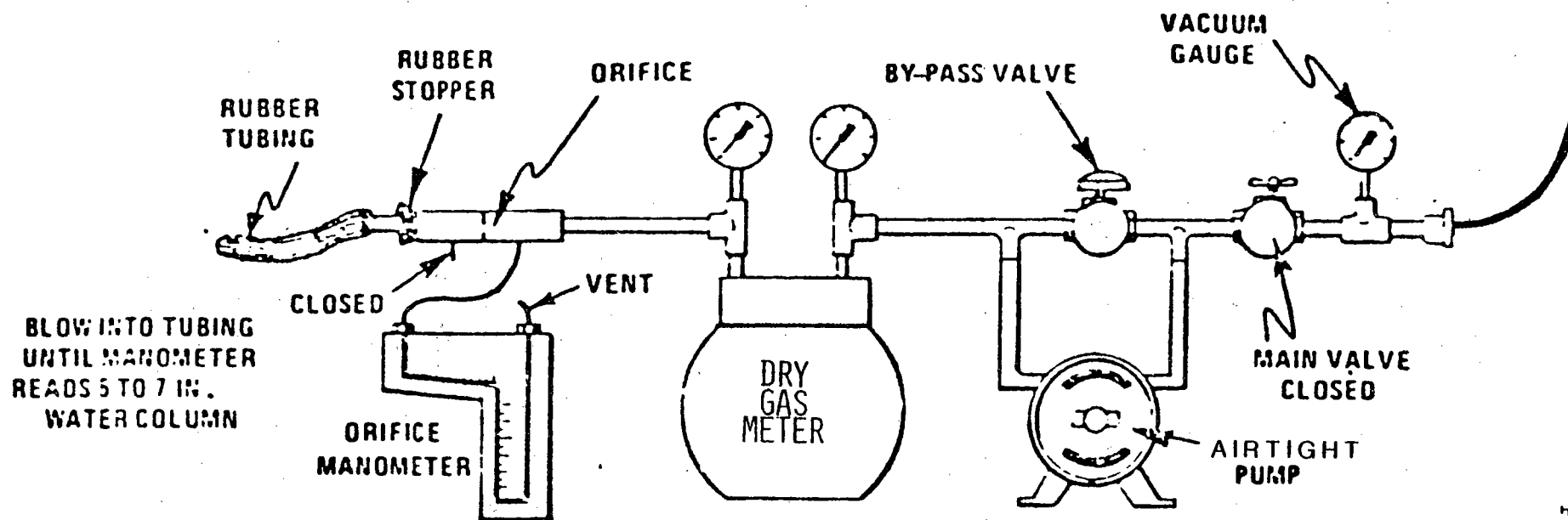


Figure 2.1 Positive leak check of metering system.

leakages within the pump. For these cases, the following leak-check procedure is suggested: make a 10-min calibration run at $0.00057 \text{ m}^3/\text{min}$ ($0.02 \text{ ft}^3/\text{min}$); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10 to get the leak rate. The leak rate should not exceed $0.00057 \text{ m}^3/\text{min}$ ($0.02 \text{ ft}^3/\text{min}$).

Initial calibration - The dry gas meter and orifice meter can be calibrated simultaneously and should be calibrated when first purchased and any time the posttest check yields a Y outside the range of the calibration factor $Y \pm 0.05Y$. A calibrated wet test meter (properly sized, with $\pm 1\%$ accuracy) should be used to calibrate the dry gas meter and the orifice meter.

The dry gas meter and the orifice meter should be calibrated in the following manner:

1. Before its initial use in the field, leak check the metering system, as described in Subsection 2.1.2. Leaks, if present, must be eliminated before proceeding.

2. Assemble the apparatus, as shown in Figure 2.2, with the wet test meter replacing the probe and impingers--that is, with the outlet of the wet test meter connected to a needle valve that is connected to the inlet side of the meter box.

3. Run the pump for 15 min with the orifice meter differential (ΔH) set at 12.7 mm (0.5 in.) H_2O to allow the pump to warm up and to permit the interior surface of the wet test meter to be wetted.

4. Adjust the needle valve so that the vacuum gauge on the meter box will read between 50 and 100 mm (2 to 4 in.) Hg during calibration.

5. Collect the information required in the forms provided (Figure 2.3A or 2.3B). Sample volumes, as shown, should be used.

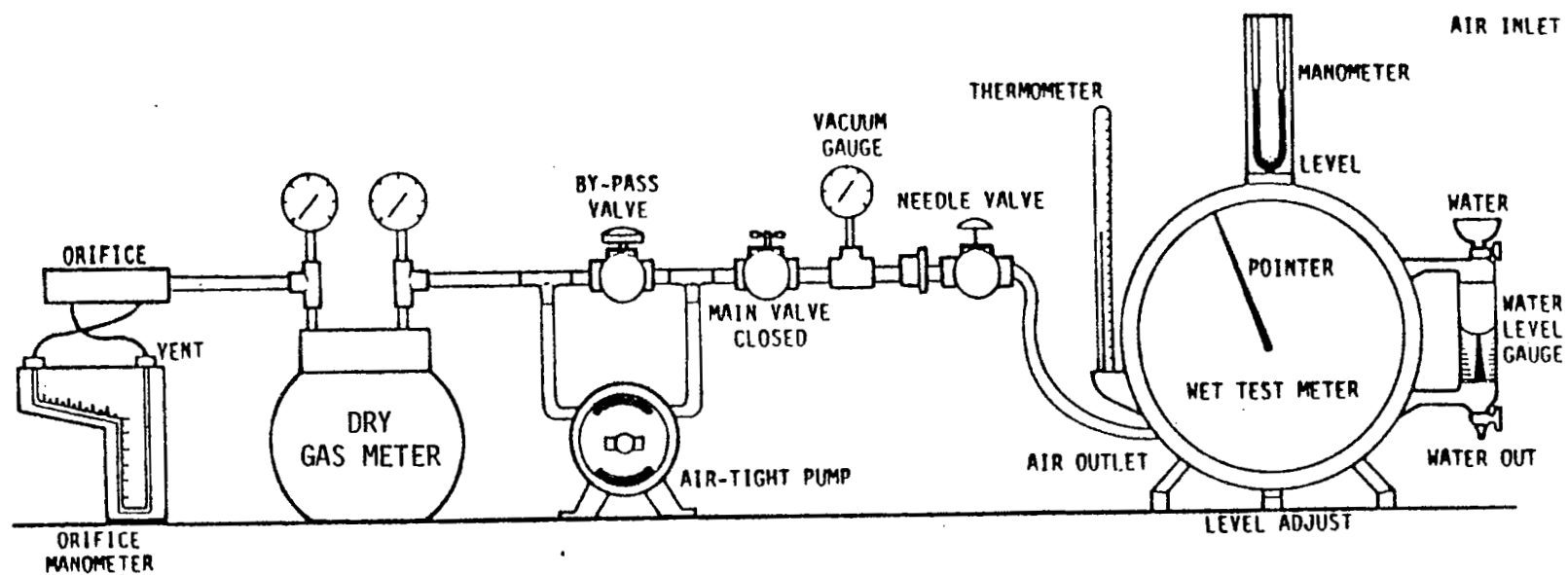


Figure 2.2 Sample meter system calibration setup.

425

Date 8/10/79

Meter box number EM-2

Barometric pressure, $P_b =$ 29.00 in. Hg Calibrated by WGD

Orifice manometer setting (ΔH), in. H_2O	Gas volume		Temperature				Time (θ), min	Y_i	$\Delta H@_i$, in. H_2O
	Wet test meter (V_w), ft^3	Dry gas meter (V_d), ft^3	Wet test meter (t_w), $^{\circ}F$	Dry gas meter					
				Inlet (t_{d_i}), $^{\circ}F$	Outlet (t_{d_o}), $^{\circ}F$	Avg ^a (t_d), $^{\circ}F$			
0.5	5	283.548 278.514	65 65	69 66	64 63	66	10.49 10.60	1.995	1.34
1.0	5								
1.5	10								
2.0	10								
3.0	10								
4.0	10								
Avg									

ΔH , in. H_2O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t + 460)}$	$\Delta H@_i = \frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460) \theta}{V_w} \right]^2$
0.5	0.0368	$\frac{5(29.00)(526)}{15.029(29.05)(525)}$	$\frac{0.317(0.5)}{29.00(526)} \left[\frac{(525)(10.60)}{5} \right]^2$
1.0	0.0737		
1.5	0.110		
2.0	0.147		
3.0	0.221		
4.0	0.294		

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d .

Figure 2.3A Dry gas meter calibration data form (English units).
(front side)

Nomenclature:

V_w = Gas volume passing through the wet test meter, ft^3 .

V_d = Gas volume passing through the dry gas meter, ft^3 .

t_w = Temperature of the gas in the wet test meter, $^{\circ}\text{F}$.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, $^{\circ}\text{F}$.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, $^{\circ}\text{F}$.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average t_{d_i} and t_{d_o} , $^{\circ}\text{F}$.

ΔH = Pressure differential across orifice, in. H_2O .

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run; tolerance $Y_i = Y \pm 0.02Y$.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all six runs; tolerance $Y = Y \pm 0.01Y$.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives $0.75 \text{ ft}^3/\text{min}$ of air at standard conditions for each calibration run, in. H_2O ; tolerance = $\Delta H@ \pm 0.15$ (recommended).

$\Delta H@$ = Average orifice pressure differential that gives $0.75 \text{ ft}^3/\text{min}$ of air at standard conditions for all six runs, in. H_2O ; tolerance = 1.84 ± 0.25 (recommended).

θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

Figure 2.3A. Dry gas meter calibration data (English units). (backside)

(426)

Date 8/10/79

Meter box number FM-2

Barometric pressure, $P_b =$ 736 mm Hg Calibrated by WGD

Orifice manometer setting (ΔH), mm H ₂ O	Gas volume		Temperature				Time (θ), min	Y_i	$\Delta H@_i$, mm H ₂ O
	Wet test meter (V_w), m ³	Dry gas meter (V_d), m ³	Wet test meter (t_w), °C	Dry gas meter					
				Inlet (t_{d_i}), °C	Outlet (t_{d_o}), °C	Avg ^a (t_d), °C			
10	0.15	25.0320 24.8800	18 18	20 19	18 17	18	29 10.20	.986	23
25	0.15								
40	0.30								
50	0.30								
75	0.30								
100	0.30								
Avg									

ΔH , mm H ₂ O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_d + \frac{\Delta H}{13.6}) (t_w + 273)}$	$\Delta H@_i = \frac{0.00117 \Delta H}{P_b (t_d + 273)} \left[\frac{(t_w + 273) \theta}{V_w} \right]^2$
10	0.7	$\frac{(0.15)(736)(291)}{(0.152)(737)(291)}$	$\frac{(0.00117)(10)}{(736)(291)} \left[\frac{(291)(10.20)}{0.152} \right]^2$
25	1.8		
40	2.94		
50	3.68		
75	5.51		
100	7.35		

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d .

Figure 2.3B Dry gas meter calibration data form (metric units).
(front side)

Nomenclature:

V_w = Gas volume passing through the wet test meter, m^3 .

V_d = Gas volume passing through the dry gas meter, m^3 .

t_w = Temperature of the gas in the wet test meter, $^{\circ}C$.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, $^{\circ}C$.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, $^{\circ}C$.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o} , $^{\circ}C$.

ΔH = Pressure differential across orifice, mm H_2O .

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run; tolerance $Y_i = Y \pm 0.02Y$.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all six runs; tolerance $Y = Y \pm 0.01Y$.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives $0.021 m^3$ of air at standard conditions for each calibration run, mm H_2O ; tolerance $\Delta H@_i = \Delta H@ \pm 3.8$ mm H_2O (recommended).

$\Delta H@$ = Average orifice pressure differential that gives $0.021 m^3$ of air at standard conditions for all six runs, mm H_2O ; tolerance $\Delta H@ = 46.74 \pm 6.3$ mm H_2O (recommended).

θ = Time of each calibration run, min.

P_b = Barometric pressure, mm Hg.

Figure 2.3B. Dry gas meter calibration data (metric units). (backside)

6. Calculate Y_i for each of the six runs, using the equation in Figure 2.3A or B under the Y_i column, and record the results on the form in the space provided.

7. Calculate the average Y for the six runs using the following equation:

$$Y = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} .$$

Record the average on Figure 2.3A or B in the space provided.

8. The dry gas meter should be cleaned, adjusted, and recalibrated, or rejected if one or more values of Y fall outside the interval $Y \pm 0.02Y$. Otherwise, the average Y (calibration factor) is acceptable and will be used for future checks and subsequent test runs.

9. Calculate $\Delta H@_i$ for each of the six runs using the equation in Figure 2.3A or B under the $\Delta H@_i$ column, and record on the form in the space provided.

10. Calculate the average $\Delta H@$ for the six runs using the following equation:

$$\Delta H@ = \frac{\Delta H@_1 + \Delta H@_2 + \Delta H@_3 + \Delta H@_4 + \Delta H@_5 + \Delta H@_6}{6} .$$

Record the average on Figure 2.3A or B in the space provided.

11. Adjust the orifice meter or reject it if $\Delta H@_i$ varies by more than ± 3.9 mm (0.15 in.) H_2O over the range of 10 to 100 mm (0.4 to 4.0 in.) H_2O . Otherwise, the average $\Delta H@$ is acceptable and will be used for subsequent test runs.

Posttest calibration check - After each field test series, conduct a calibration check of the metering system, as in Subsection 2.1.2, except for the following variations:

1. Three calibration runs at a single intermediate orifice meter setting may be used with the vacuum set at the maximum value reached during the test series. The single intermediate

(429)

orifice meter setting should be based on the previous field test. A valve must be inserted between the wet test meter and the inlet of the metering system to adjust the vacuum.

2. If a temperature-compensating dry gas meter was used, the calibration temperature for the dry gas meter must be within $\pm 6^{\circ}\text{C}$ (10.8°F) of the average meter temperature during the test series.

3. Use Figure 2.4A or 2.4B, and record the required information.

If the calibration factor Y deviates by $<5\%$ from the initial calibration factor Y (determined in Subsection 2.1.2), then the dry gas meter volumes obtained during the test series are acceptable. If Y deviates by $>5\%$, recalibrate the metering system (as in Subsection 2.1.2), and use whichever meter coefficient (initial or recalibrated) that yields the lowest gas volume for each test run.

Alternate procedures--for example, using the orifice meter coefficients--may be used, subject to the approval of the administrator.

2.2 Temperature Gauges

2.2.1 Impinger Thermometer - The thermometer used to measure the temperature of the gas stream exiting the impinger train should initially be compared with a mercury-in-glass thermometer which meets ASTM E-1 No. 63C or 63F specifications. The procedure is as follows:

1. Place both the reference thermometer and the test thermometer in an ice bath. Compare readings after they both stabilize.

2. Remove the thermometers from the bath and allow both to come to room temperature. Again, compare readings after they both stabilize.

3. Accept the test thermometer if its reading agrees within $\pm 1^{\circ}\text{C}$ (2°F) of the reference thermometer reading at both temperatures. If the difference is greater than $\pm 1^{\circ}\text{C}$ (2°F),

Test number AB1-3 Date 9/13/79 Meter box number FM-7 Plant Acme Power Plant
 Barometric pressure, $P_b = 28.72$ in. Hg Dry gas meter number FM-7 Pretest Y 0.986

Orifice manometer setting, (ΔH), in. H ₂ O	Gas volume		Temperature				Time (Θ), min	Vacuum setting, in. Hg	Y _i	Y _i	$\frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6})(t_w + 460)}$
	Wet test meter (V _w), ft ³	Dry gas meter (V _d), ft ³	Wet test meter (t _w), °F	Dry gas meter							
				Inlet (t _{d_i}), °F	Outlet (t _{d_o}), °F	Average _a (t _d), °F					
1.41	.10	886.544 876.321	72	83	75	79	13.35	3	0.987		$\frac{10(28.72)(79 + 460)}{10.223(28.72 + \frac{1.41}{13.6})(72 + 460)}$
	10										
	10										
Y =											

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d
 where

V_w = Gas volume passing through the wet test meter, ft³.

V_d = Gas volume passing through the dry gas meter, ft³.

t_w = Temperature of the gas in the wet test meter, °F.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, °F.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, °F.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o} , °F.

ΔH = Pressure differential across orifice, in. H₂O.

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs;
 tolerance = pretest $Y \pm 0.05Y$.

P_b = Barometric pressure, in. Hg.

θ = Time of calibration run, min.

Figure 2.4A Posttest dry gas meter calibration data form (English units).

Test number P31-3 Date 9/13/79 Meter box number FM-7 Plant AONE Power Plant
 Barometric pressure, P_b = 730 mm Hg Dry gas meter number FM-7 Pretest Y 0.993

Orifice manometer setting, (ΔH), mm H ₂ O	Gas volume		Temperature				Time (θ), min	Vacuum setting, mm Hg	Y _i	Y _i
	Wet test meter (V _w), m ³	Dry gas meter (V _d), m ³	Wet test meter (t _w), °C	Dry gas meter						V _w P _b (t _d + 273)
				Inlet (t _d _i), °C	Outlet (t _d _o), °C	Average ^a (t _d), °C				V _d P _b + $\frac{\Delta H}{13.6} t_w + 273$
36	.3	26.1742 25.5730	21	23.5	21.5	22.5	13.5	7.5	0.990	$\frac{36 \times 730 \times (21.8 + 273)}{0.3012 \times (730 + \frac{36}{13.6} \times 21.8 + 273)}$
	.3									
	.3									
Y =										

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d where

V_w = Gas volume passing through the wet test meter, m³.

V_d = Gas volume passing through the dry gas meter, m³.

t_w = Temperature of the gas in the wet test meter, °C.

t_{di} = Temperature of the inlet gas of the dry gas meter, °C.

t_{do} = Temperature of the outlet gas of the dry gas meter, °C.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{di} and t_{do} , °C.

ΔH = Pressure differential across orifice, mm H₂O.

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs;
tolerance = pretest Y $\pm 0.05Y$.

P_b = Barometric pressure, mm Hg.

θ = Time of calibration run, min.

Figure 2.4B Posttest meter calibration data form (metric units).

the thermometer should be adjusted and recalibrated until the criteria are met, or it should be rejected.

4. Prior to each field trip compare the room temperature with the meter thermometer and the mercury-in-glass thermometer. If the readings are not within $\pm 2^{\circ}\text{C}$ (4°F), the meter thermometer should be replaced or recalibrated.

2.2.2 Dry Gas Thermometers - The thermometers used to measure the metered gas sample temperature should initially be compared with a mercury-in-glass thermometer as above, using a similar procedure.

1. Place the reference and the test thermometers in a hot water bath maintained at 40° to 50°C (104° to 122°F). Compare the readings after both stabilize.

2. Allow both thermometers to come to room temperature. Compare readings after the thermometers stabilize.

3. Accept the test thermometer if its reading agrees within 3°C (5.4°F) of the reference thermometer reading at both temperatures. If not, either the thermometer should be adjusted and recalibrated or a temperature correction factor should be marked on the thermometer where it is readily visible to the operator. When the factor is used, it must be noted on the pretest sampling check form (Figure 2.5) and in the calibration log.

4. Compare the temperatures prior to each field trip at room temperature with the thermometer as part of the meter system. If the readings or corrected values are not within $\pm 6^{\circ}\text{C}$ (10.8°F) of the mercury-in-glass thermometer value, the meter thermometer should be replaced or recalibrated.

2.3 Barometer

The field barometer should be adjusted initially and before each test series to agree within ± 2.5 mm (0.1 in.) Hg of the mercury-in-glass barometer or with the pressure reported by a nearby National Weather Service Station. Correction for elevation difference between the station and the sampling point should be applied at a rate of -2.5 mm Hg/30 m (0.1 in. Hg/100 ft). Record results on the pretest sampling check form (Figure 2.1).

Date January 12, 1980 Calibrated by B. C. Blagun
Meter box number FM-1 $\Delta H@$ 1.41

Dry Gas Meter*

Pretest calibration factor 0.986 (within 2% of the average factor for each calibration run)

Impinger Thermometer

Was a pretest temperature correction used? yes ☒ no
If yes, temperature correction _____ (within 2°C (4°F) of reference value)

Dry Gas Meter Thermometer

Was a pretest temperature correction made? yes ☒ no
If yes, temperature correction _____ (within 6°C (10.8°F) of reference value)

Barometer

Was the pretest field barometer reading correct? ☒ yes ☐ no

Stack Gas Temperature Sensor (if required)*

Was a temperature sensor required for moisture determination purposes? yes ☒ no

Was a pretest temperature correction used? yes ☒ no
If yes, temperature correction _____ (within $\pm 1^\circ\text{C}$ over the entire range)

Did the temperature sensor agree with the reference thermometer (within $\pm 1^\circ\text{C}$ (2°F) over the range of 10° to 82°C (50° to 180°F))? ☒ yes ☐ no

*Most significant items/parameters to be checked.

Figure 2.5 Pretest sampling checks.

4/34

2.4 Trip Balance

The trip balance should be calibrated initially by using Class-S standard weights and should be within ± 0.5 g of the standard weight. Adjust or return the balance to the manufacturer if limits are not met.

2.5 Stack Gas Temperature Sensor

The stack gas temperature must be accurately determined when the stack is suspected of being saturated or having water droplets. Therefore proper calibration of the stack gas temperature sensor is important for this method. Upon receipt, the sensor should be calibrated over the entire range. An ASTM E-1 No. 3C or 3F thermometer should be used as the reference temperature. The initial and, as required, recalibration procedure is as follows:

1. Place both the temperature sensor and the reference thermometer in water or in a controlled-temperature atmosphere.
2. Record both temperatures after each has stabilized for 30 s. Increase the temperature in increments of about 6°C (10°F), taking readings over the entire range (10 - 82°C (50 - 180°F)).
3. Both values should agree within $\pm 1^{\circ}\text{C}$ (2°F). If not, the temperature sensor should be adjusted if possible. However, if the values are off by a constant factor over the entire range, a correction factor may be used.

After each field use, the temperature sensor calibration should be checked. The procedure for the check is as follows:

1. Check the temperature sensor with the reference thermometer at a temperature within $\pm 5^{\circ}\text{C}$ (10°F) of the average stack temperature. If the values agree within $\pm 2^{\circ}\text{C}$ (4°F), then the pretest calibration is acceptable.

2. When the above agreement is not met, the temperature sensor should be recalibrated at a temperature within $\pm 2^{\circ}\text{C}$ (4°F) of the average stack temperature, and a correction factor should be determined with the reference thermometer. The difference

between the temperature sensor and the reference thermometer should be used to correct the average stack temperature for calculation purposes. Also, a complete recalibration of the temperature sensor is suggested.

Table 2.1 ACTIVITY MATRIX FOR EQUIPMENT CALIBRATION REQUIREMENTS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Wet test meter	Capacity of at least $3.4 \text{ m}^3/\text{h}$ ($120 \text{ ft}^3/\text{h}$) and accuracy within $\pm 1\%$	Calibrate initially and then yearly by the liquid displacement technique; see Subsec 2.1.1	Adjust until specifications are met, or return to manufacturer
Dry gas meter	$Y_1 = Y \pm 0.02Y$ at a flow rate of $0.02\text{--}0.03 \text{ m}^3/\text{min}$ ($0.66\text{--}1 \text{ ft}^3/\text{min}$)	Calibrate vs. wet test meter initially to agree, and when the posttest check is not within $Y \pm 0.05Y$	Repair or replace, and then recalibrate
Thermometers	Impinger thermometer $\pm 1^\circ\text{C}$ (2°F); dry gas meter thermometer $\pm 3^\circ\text{C}$ (5.4°F) over range	Calibrate each initially as a separate component against a mercury-in-glass thermometer; before each field trip, compare each as part of the train with the mercury-in-glass thermometer	Adjust, determine a constant correction factor, or reject
Barometer	$\pm 2.5 \text{ mm}$ (0.1 in.) Hg of the mercury-in-glass barometer	Calibrate initially using mercury-in-glass barometer; check before and after each field test	Adjust to agree with certified barometer
Stack gas temperature sensor for moisture determination	Pretest calibration $\pm 1^\circ\text{C}$ (2°F) over range; posttest check $\pm 2^\circ\text{C}$ (4°F)	Calibrate initially over the range with an ASTM reference thermometer; after each field test, make a single-point calibration check	Adjust to agree with reference thermometer; use a constant correction factor, or reject; posttest data corrected for calculation purposes

3. 2. 1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. 12. 13. 14. 15. 16. 17. 18. 19. 20. 21. 22. 23. 24. 25. 26. 27. 28. 29. 30. 31. 32. 33. 34. 35. 36. 37. 38. 39. 40. 41. 42. 43. 44. 45. 46. 47. 48. 49. 50. 51. 52. 53. 54. 55. 56. 57. 58. 59. 60. 61. 62. 63. 64. 65. 66. 67. 68. 69. 70. 71. 72. 73. 74. 75. 76. 77. 78. 79. 80. 81. 82. 83. 84. 85. 86. 87. 88. 89. 90. 91. 92. 93. 94. 95. 96. 97. 98. 99. 100. 101. 102. 103. 104. 105. 106. 107. 108. 109. 110. 111. 112. 113. 114. 115. 116. 117. 118. 119. 120. 121. 122. 123. 124. 125. 126. 127. 128. 129. 130. 131. 132. 133. 134. 135. 136. 137. 138. 139. 140. 141. 142. 143. 144. 145. 146. 147. 148. 149. 150. 151. 152. 153. 154. 155. 156. 157. 158. 159. 160. 161. 162. 163. 164. 165. 166. 167. 168. 169. 170. 171. 172. 173. 174. 175. 176. 177. 178. 179. 180. 181. 182. 183. 184. 185. 186. 187. 188. 189. 190. 191. 192. 193. 194. 195. 196. 197. 198. 199. 200. 201. 202. 203. 204. 205. 206. 207. 208. 209. 210. 211. 212. 213. 214. 215. 216. 217. 218. 219. 220. 221. 222. 223. 224. 225. 226. 227. 228. 229. 230. 231. 232. 233. 234. 235. 236. 237. 238. 239. 240. 241. 242. 243. 244. 245. 246. 247. 248. 249. 250. 251. 252. 253. 254. 255. 256. 257. 258. 259. 260. 261. 262. 263. 264. 265. 266. 267. 268. 269. 270. 271. 272. 273. 274. 275. 276. 277. 278. 279. 280. 281. 282. 283. 284. 285. 286. 287. 288. 289. 290. 291. 292. 293. 294. 295. 296. 297. 298. 299. 300. 301. 302. 303. 304. 305. 306. 307. 308. 309. 310. 311. 312. 313. 314. 315. 316. 317. 318. 319. 320. 321. 322. 323. 324. 325. 326. 327. 328. 329. 330. 331. 332. 333. 334. 335. 336. 337. 338. 339. 340. 341. 342. 343. 344. 345. 346. 347. 348. 349. 350. 351. 352. 353. 354. 355. 356. 357. 358. 359. 360. 361. 362. 363. 364. 365. 366. 367. 368. 369. 370. 371. 372. 373. 374. 375. 376. 377. 378. 379. 380. 381. 382. 383. 384. 385. 386. 387. 388. 389. 390. 391. 392. 393. 394. 395. 396. 397. 398. 399. 400. 401. 402. 403. 404. 405. 406. 407. 408. 409. 410. 411. 412. 413. 414. 415. 416. 417. 418. 419. 420. 421. 422. 423. 424. 425. 426. 427. 428. 429. 430. 431. 432. 433. 434. 435. 436. 437. 438. 439. 440. 441. 442. 443. 444. 445. 446. 447. 448. 449. 450. 451. 452. 453. 454. 455. 456. 457. 458. 459. 460. 461. 462. 463. 464. 465. 466. 467. 468. 469. 470. 471. 472. 473. 474. 475. 476. 477. 478. 479. 480. 481. 482. 483. 484. 485. 486. 487. 488. 489. 490. 491. 492. 493. 494. 495. 496. 497. 498. 499. 500. 501. 502. 503. 504. 505. 506. 507. 508. 509. 510. 511. 512. 513. 514. 515. 516. 517. 518. 519. 520. 521. 522. 523. 524. 525. 526. 527. 528. 529. 530. 531. 532. 533. 534. 535. 536. 537. 538. 539. 540. 541. 542. 543. 544. 545. 546. 547. 548. 549. 550. 551. 552. 553. 554. 555. 556. 557. 558. 559. 560. 561. 562. 563. 564. 565. 566. 567. 568. 569. 570. 571. 572. 573. 574. 575. 576. 577. 578. 579. 580. 581. 582. 583. 584. 585. 586. 587. 588. 589. 590. 591. 592. 593. 594. 595. 596. 597. 598. 599. 600. 601. 602. 603. 604. 605. 606. 607. 608. 609. 610. 611. 612. 613. 614. 615. 616. 617. 618. 619. 620. 621. 622. 623. 624. 625. 626. 627. 628. 629. 630. 631. 632. 633. 634. 635. 636. 637. 638. 639. 640. 641. 642. 643. 644. 645. 646. 647. 648. 649. 650. 651. 652. 653. 654. 655. 656. 657. 658. 659. 660. 661. 662. 663. 664. 665. 666. 667. 668. 669. 670. 671. 672. 673. 674. 675. 676. 677. 678. 679. 680. 681. 682. 683. 684. 685. 686. 687. 688. 689. 690. 691. 692. 693. 694. 695. 696. 697. 698. 699. 700. 701. 702. 703. 704. 705. 706. 707. 708. 709. 710. 711. 712. 713. 714. 715. 716. 717. 718. 719. 720. 721. 722. 723. 724. 725. 726. 727. 728. 729. 730. 731. 732. 733. 734. 735. 736. 737. 738. 739. 740. 741. 742. 743. 744. 745. 746. 747. 748. 749. 750. 751. 752. 753. 754. 755. 756. 757. 758. 759. 760. 761. 762. 763. 764. 765. 766. 767. 768. 769. 770. 771. 772. 773. 774. 775. 776. 777. 778. 779. 780. 781. 782. 783. 784. 785. 786. 787. 788. 789. 790. 791. 792. 793. 794. 795. 796. 797. 798. 799. 800. 801. 802. 803. 804. 805. 806. 807. 808. 809. 810. 811. 812. 813. 814. 815. 816. 817. 818. 819. 820. 821. 822. 823. 824. 825. 826. 827. 828. 829. 830. 831. 832. 833. 834. 835. 836. 837. 838. 839

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3.0 PRESAMPLING OPERATIONS

The quality assurance activities for presampling operations are summarized in Table 3.1 at the end of this section. See Section 3.0, the introduction to this Handbook, for details on preliminary site visits.

3.1 Apparatus Check and Calibration

A pretest check will have to be made on most of the sampling apparatus. Figure 3.1 should be used as a pretest operations and packing list. An inquiry must be made as to whether the stack gas is saturated or has water droplets.

3.1.1 Sampling Train - The specifications of the Method 4 sampling train used by the EPA are given in Figure 1.1. Commercial models of this system are available. Each individual or fabricated train must be in compliance with the specifications of the Reference Method, Section 3.3.10.

3.1.2 Probe - Clean the probe internally by brushing first with tap water, then with deionized distilled water, and finally with acetone; allow it to dry in the air. In extreme cases, the probe liner can be cleaned with stronger reagents. In either case, the objective is to leave the probe liner free from contaminants. The probe's heating system should be checked to see that it is operating properly. The probe should be sealed at the inlet or tip and checked for leaks at a vacuum of 380 mm (15 in.) Hg, and the probe must be leak free under these conditions.

3.1.3 Impinger and Glass Connections - All glassware should be cleaned first with detergent and thoroughly rinsed with tap water and then with deionized distilled water. All glassware should be visually inspected for cracks or breakage and then repaired or discarded if defective.

3.1.4 Pump - The vacuum pump should be serviced as recommended by the manufacturer, or every 3 mo, or upon erratic behavior (nonuniform or insufficient pumping action). Check oiler jars, if used, every 10 tests.

434

| Apparatus check | Acceptable | | Quantity required | Ready | | Packed and loaded | |
|--|-------------------------------------|----|--------------------|-------------------------------------|----|-------------------------------------|----|
| | Yes | No | | Yes | No | Yes | No |
| <u>Probe type</u> | | | | | | | |
| Borosilicate glass <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 3 | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Quartz glass _____ | | | | | | | |
| Other _____ | | | | | | | |
| Heater and leak checked* <input checked="" type="checkbox"/> | | | | | | | |
| <u>Filter</u> | | | | | | | |
| In-stack <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 1 Box | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Out-stack _____ | | | | | | | |
| Glass wool _____ | | | | | | | |
| Other _____ | | | | | | | |
| <u>Condenser</u> | | | | | | | |
| Impingers <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 5 L.S.
16 mm ID | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Other _____ | | | | | | | |
| <u>Cooling System</u> | | | | | | | |
| Ice bath <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 3 | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Other _____ | | | | | | | |
| <u>Metering System</u> | | | | | | | |
| Vacuum gauge <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 2 | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Checked* <input checked="" type="checkbox"/> | | | | | | | |
| Pump <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 2 | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Leak checked* <input checked="" type="checkbox"/> | | | | | | | |
| Thermometers <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | | | | <input checked="" type="checkbox"/> | |
| Calibrated* <input checked="" type="checkbox"/> | | | | | | | |
| Dry gas meter <input checked="" type="checkbox"/> | <input checked="" type="checkbox"/> | | 3 | <input checked="" type="checkbox"/> | | <input checked="" type="checkbox"/> | |
| Calibrated* <input checked="" type="checkbox"/> | | | | | | | |
| Other _____ | | | | | | | |

*Most significant items/parameters to be checked.

Figure 3.1 Pretest preparation checklist. (continued)

Figure 3.1 (continued)

| Apparatus check | Acceptable | | Quantity required | Ready | | Packed and loaded | |
|----------------------------------|------------|----|-------------------|-------|----|-------------------|----|
| | Yes | No | | Yes | No | Yes | No |
| <u>Barometer</u> | | | | | | | |
| Mercury <u>✓</u> | ✓ | | 1 | ✓ | | ✓ | |
| Aneroid <u> </u> | | | | | | | |
| Other <u> </u> | | | | | | | |
| Calibrated* <u>✓</u> | | | | | | | |
| <u>Quantitative Instrument</u> | | | | | | | |
| Graduated cylinder <u>✓</u> | ✓ | | 3 | ✓ | | ✓ | |
| Trip balance <u>✓</u> | ✓ | | 2 | ✓ | | ✓ | |
| Calibrated* <u>YES</u> | | | | ✓ | | ✓ | |
| <u>Stack Temperature Sensor*</u> | | | | | | | |
| Type <u>THERMOCOUPLE</u> | ✓ | | 2 | ✓ | | ✓ | |
| Calibrated <u>YES</u> | | | | | | | |

*Most significant items/parameters to be checked.

(1/11)

3.1.5 Dry Gas Meter - A dry gas meter calibration check should be made using the procedure in Section 3.3.2.

3.1.6 Silica Gel - Either dry the used silica gel at 175°C (350°F) or use fresh silica gel and weigh several 200- to 300-g portions in airtight containers to the nearest 0.5 g. Record the total weight (silica gel plus container) on each container.

3.1.7 Thermometers - The thermometers should be compared to the mercury-in-glass reference thermometer at ambient temperature (Subsection 2.2.1 of Section 3.3.2).

3.1.8 Barometer - The field barometer should be compared with the mercury-in-glass barometer or the weather station reading prior to each field trip (Section 3.3.2).

3.1.9 Stack Gas Temperature Sensor - A specially calibrated temperature sensor is required if the stack gas is saturated or has water droplets present. The sensor should be calibrated against a reference thermometer (Section 3.3.2).

3.1.10 Water - It is recommended, but not required, that 100 ml of deionized distilled water conforming to ASTM D1193-74 type 3 be used in each of the first two impingers.

3.2 Equipment Packing

The accessibility, condition, and functioning of measurement devices in the field depend on careful packing and on the care of movement on site. Equipment should be packed to withstand severe treatment during shipping and field handling operations. One major consideration in shipping cases is the construction materials. The following containers are suggested, but are not mandatory.

3.2.1 Probe - Seal the inlet and outlet of the probe and then wrap with polyethylene or other suitable material to protect the probe from breakage. An ideal container is a wooden case (or equivalent) lined with foam material and with separate compartments to hold individual probes. The case should have handles or eye-hooks that can withstand hoisting and that will be rigid enough to prevent bending or twisting during shipping and handling.

(442)

3.2.2 Impingers, Connectors, and Assorted Glassware - All impingers and glassware should be packed in rigid containers and protected by polyethylene packing material or other suitable material. Individual compartments for glassware will help to organize and protect each piece and simplify inventorying.

3.2.3 Volumetric Glassware - A sturdy case lined with foam material can contain drying tubes and assorted volumetric glassware.

3.2.4 Meter Box - The meter box--which contains the manometers, orifice meter, vacuum gauge, pump, dry gas meter, and thermometers--should be packed in a shipping container unless its housing is sufficient to protect components during travel. Pump oil sump and oiler jars should be drained to prevent fouling of the components during shipment. Additional pump oil should be packed if oil is required. It is advisable to carry a spare meter box in case of failure.

3.2.5 Wash Bottles and Storage Containers - Storage containers and miscellaneous glassware should be packed in a rigid foam-lined container.

(4/4)

Table 3.1 ACTIVITY MATRIX FOR PRESAMPLING PREPARATIONS

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|---|---|---|---|
| Probe | <p>1. Probe liner free of contaminants and constructed of borosilicate glass, quartz, or equivalent; metal liners must be approved by administrator</p> <p>2. Probe leak free at 380 mm (15 in.) Hg</p> <p>3. Probe that prevents moisture condensation</p> | <p>1. Clean probe internally by brushing using tap water, then deionized distilled water, and finally acetone; air dry before test</p> <p>2. Visually check before test</p> <p>3. Check heating system initially and when moisture cannot be prevented during testing</p> | <p>1. Repeat cleaning procedure, and reassemble</p> <p>2. Replace</p> <p>3. Repair or replace</p> |
| Impingers, filter holders, and glass containers | Clean and free of breaks, cracks, leaks, etc. | Clean with detergent and tap water, then deionized distilled water | Repair or discard |
| Pump | Sampling rate of about 0.02-.03 m ³ /min (0.66-1 ft ³ /min) up to 380 mm (15 in.) Hg vacuum at pump inlet | Service every 3 mo or upon erratic behavior; check oiler jars every 10 tests | Repair or return to manufacturer |
| Dry gas meter | Readings within $\pm 2\%$ average calibration factor; clean | Calibrate according to Sec 3.3.2 check for excess oil | As above |
| Thermometers | Readings within $\pm 2^{\circ}\text{C}$ (4°F) of mercury-in-glass thermometer | Compare with mercury-in-glass thermometer at room temperature prior to each field test | Replace or recalibrate |
| Barometer | Readings within 2.5 mm (0.1 in.) Hg | Compare with mercury-in-glass barometer or value reported by nearby National Weather Station corrected for elevation prior to each field test | As above |

(continued)

Table 3.1 (continued)

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|---|---|--|------------------------------------|
| Stack gas temperature sensor for moisture determination | $\pm 1^{\circ}\text{C}$ (2°F) over range of 10° to 80°C (50° to 180°F) | Compare against ASTM reference thermometer | As above |
| Water | Deionized distilled; ASTM-D1193-74 type 3 | Run blank evaporations prior to field use to eliminate high solids (only required if impinger contents to be analyzed) | Redistill or replace |
| Silica gel | Indicating type, size 6 to 16 mesh; dry used gel at 175°C (350°F) for at least 2 h; weigh 200 g portion to nearest 0.5 g; record the weight | Prior to each field test, observe drying time if appropriate; check weighings | Repeat procedure |
| <u>Package Equipment for Shipment</u> | | | |
| Probe | Packed in rigid container and protected by polyethylene foam | Prior to each shipment, check packing of equipment | Repack |
| Impingers, containers, and assorted glassware | Packed in rigid container and protected by polyethylene foam | As above | As above |
| Meter box | Meter box case and/or additional material to protect train components; pack spare meter box | As above | As above |

4.0 ON-SITE MEASUREMENTS

The on-site activities include transporting the equipment to the test site, unpacking and assembling the equipment, making duct measurements, determining whether the stack gas is saturated or has water droplets, charging the impingers, obtaining a sample, and recording data. Table 4.1 at the end of this section summarizes the on-site quality assurance activities and Figure 4.1 is an on-site measurement checklist. Blank data forms are in Section 3.3.12 for the convenience of the Handbook user.

4.1 Handling Equipment

The most efficient means of transporting or moving the equipment from ground level to the sampling site should be decided during the preliminary site visit or through prior correspondence to minimize damage to the test equipment or injury to test personnel. A "laboratory" area should be designated for assembling the sampling train, placing the filter in the filter holder, charging the impingers, recovering the sample, and documenting the results; this area should be clean and should be free of excessive drafts.

4.2 Sampling

The on-site sampling includes addition of the water and silica gel to the impingers; setup of the sampling train; connection to the electrical service; preparation of the probe (leak check of entire sampling train and addition of particulate filter); insertion of the probe into stack; sealing of the port; check of the probe temperature; and sampling and recording the data (Figure 4.2). A final leak check of the train is mandatory after sampling.

4.2.1 Preliminary Measurements and Setup - The sampling site should be selected in accordance with Method 1. If this is impossible due to duct configuration or other reasons, the site should be approved by the administrator. A 115 V, 30-A

Procedure used: Reference ✓ Approximate _____

Reference Method

Conducted simultaneously with pollutant emission test? yes

Impingers properly placed?* yes

Impinger content: 1st 100 ml H₂O 2nd 100 ml H₂O 3rd empty

4th 200g Silica gel Modifications _____

Cooling System: Crushed ice ✓ Other _____

Sampling time per point 5 min

Probe heater (if applicable) on? yes Temp 250°F

Crushed ice in ice bath? yes

Leak check? (optional) yes Leakage rate 0.0

Sampling rate constant (within 10%)* yes

All data properly recorded?* yes

Posttest leak check?* (mandatory) yes

Leakage rate* 0.0

Analysis - Impinger Content

Method: Volumetric ✓ Gravimetric _____

Measurement of volume of water condensed:

Graduated cylinder yes Other _____

Measurement of silica gel: Balance ✓ Other _____

Color of silica gel? blue Condition very little moisture

All analytical data properly recorded? yes

*Most significant items/parameters to be checked.

Figure 4.1 On-site measurement checklist

Plant Acme Power Plant Probe material Glass
 Location Boiler No. 1 Outlet Sample box number RG-3
 Operator MB Meter box number FM-2
 Date 8/10/79 Meter ΔH_0 1.41
 Run number APP-1 Meter cal. (Y) 1.016
 Ambient temperature 68°F Final leak rate 0.00
 Barometric pressure 29.00 Vacuum during leak check 2.0
 Probe length μ (ft) 8.54 Thermometer number BE-5
 Static pressure -10.0 in H₂O

| | Impinger volume, ml | Silica gel weight, g |
|---------|---------------------|----------------------|
| Final | 271 | 215.0 |
| Initial | 200 | 203.5 |

| Traverse point number | Sampling time (θ), min | Stack temperature, $^{\circ}\text{C}$ ($^{\circ}\text{F}$) | Pressure differential across orifice meter (ΔH), mm (in.) H ₂ O | Meter reading gas sample volume, m ³ (ft ³) | ΔV_m , * m ³ (ft ³) | Gas sample temperature at dry gas meter | | Temperature of gas leaving condenser or last impinger, $^{\circ}\text{C}$ ($^{\circ}\text{F}$) |
|-----------------------|---------------------------------|--|--|--|--|--|--|--|
| | | | | | | Inlet (T _{m_{in}}) $^{\circ}\text{C}$ ($^{\circ}\text{F}$) | Outlet (T _{m_{out}}) $^{\circ}\text{C}$ ($^{\circ}\text{F}$) | |
| Start | 0 | 129 | — | 517.321 | — | — | — | — |
| 1 | 5 | 133 | 1.0 | 519.95 | 2.63 | 68 | 68 | 68 |
| 2 | 10 | 136 | 1.0 | 522.59 | 2.64 | 72 | 69 | 65 |
| 3 | 15 | 137 | 1.0 | 525.21 | 2.62 | 76 | 70 | 65 |
| 4 | 20 | 137 | 1.0 | 527.85 | 2.64 | 78 | 71 | 66 |
| 5 | 25 | 137 | 1.0 | 530.49 | 2.64 | 80 | 72 | 66 |
| 6 | 30 | 138 | 1.0 | 533.10 | 2.61 | 84 | 73 | 65 |
| 7 | 35 | 138 | 1.0 | 535.73 | 2.63 | 86 | 75 | 65 |
| 8 | 40 | 137 | 1.0 | 538.36 | 2.63 | 88 | 75 | 66 |
| 9 | 45 | 137 | 1.0 | 541.00 | 2.64 | 90 | 76 | 67 |
| 10 | 50 | 135 | 1.0 | 543.62 | 2.62 | 93 | 78 | 67 |
| 11 | 55 | 130 | 1.0 | 546.25 | 2.63 | 95 | 79 | 68 |
| 12 | 60 | 129 | 1.0 | 548.86 | 2.61 | 96 | 80 | 68 |
| Total | 60 | | | | | | | |
| Average | | 134.8 | 1.0 | | 2.628 | | 78.8 | |

* Acceptable $\Delta V_m = 0.9 < \frac{V_{\text{final}} - V_{\text{initial}}}{\text{number of points}} < 1.1$ 2.37 to 2.89.

Figure 4.2 Method 4 field and sample recovery data form.

electrical supply is necessary to operate the standard sampling train. A minimum of eight traverse points should be used for rectangular stacks having equivalent diameters <0.61 m (<24 in.), and a minimum of 12 should be used for all other stacks unless otherwise specified by the administrator. Record all data on the traverse point location form shown in Section 3.0 (introduction to this volume). These measurements will be used to locate the sampling probe during preliminary measurements and actual sampling.

Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of the probe.

Select a total sampling time so that a minimum gas volume of 0.60 m^3 (21 sft^3) can be collected at a constant rate of $\leq 0.021 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{m}$). The rate can be limited by selecting a pressure drop (ΔH) which is $< \Delta H_0$ for the orifice meter.

Note: If moisture saturated or droplet-laden gas streams are suspected, two calculations of the moisture content of the stack gas should be made--one using a value based on the saturated conditions (Equation 4-1) and another using the results of the impinger analysis. The lower of these two B_{ws} values should be considered correct.

To determine the moisture content in moisture saturated or droplet-laden gas streams, attach a temperature sensor capable of measuring $\pm 1^\circ\text{C}$ (2°F) to the probe; measure the stack gas temperature at each traverse point during the traverse; measure the absolute stack pressure. Determine the moisture percentage, either by:

1. Using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or

2. Using saturation vapor pressure (S.V.P.) Tables 6.1A and 6.1B of Section 3.3.6 and Equation 4-1.

2/15

$$B_{ws} = \frac{S.V.P.}{P_{bar} + \frac{P_{static}}{13.6}} \quad \text{Equation 4-1}$$

where

B_{ws} = water vapor in the gas stream, proportion by volume

S.V.P. = saturated vapor pressure of water at average stack temperature, mm (in.) Hg

P_{bar} = barometric pressure, mm (in.) Hg, and

P_{static} = static pressure of the stack, mm (in.) H_2O .

If the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods approved by the administrator should be used.

The stack gas can be checked for saturation with wet and dry bulb thermometers. When the stack is saturated, the wet and dry bulb temperatures are the same. This will not, however, check for the presence of water droplets.

4.2.2 Condenser Preparation - Place known volumes of water in the first and second impingers; generally, 100 ml in each impinger is adequate. Immerse the tips of the impinger tubes at least 13.0 mm (0.5 in.) in the water. The third impinger should be left dry to trap any entrained water droplets. Place a known amount of silica gel in the fourth impinger; generally, 200 g is sufficient. Record the amount of water and silica gel placed in the impingers. If the stack temperature is high $>400^{\circ}C$ ($752^{\circ}F$), a lower sampling rate may be necessary to maintain the temperature leaving the fourth impinger at $\leq 20^{\circ}C$ ($\leq 68^{\circ}F$).

Alternatively, each impinger and its contents can be weighed to the nearest 0.5 g. Record these weights on the analytical data form (Figure 4.3) for determining the amount of water condensed.

4.2.3 Sampling Train Assembly - Assemble the sampling train as shown in Figure 1.1, and perform the following:

1. Adjust the probe heater to operating temperature, and place crushed ice and water around the impingers.

(48)

Plant ACME Power Plant
Date 8-10-79
Run number APP-1

| | Volume of liquid water collected | |
|------------------------|----------------------------------|----------------------|
| | Impinger volume, ml | Silica gel weight, g |
| Final | 271 | 215.0 |
| Initial | 200 | 203.5 |
| Liquid collected | 71 | 11.5 |
| Total volume collected | | g* 825ml |

* Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml):

$$\frac{\text{Increase, g}}{1 \text{ g/ml}} = \text{water volume, ml.}$$

Figure 4.3 Method 4 analytical data form.

2. Leak check the sampling train just prior to use by disconnecting the probe from the first impinger or (if applicable, from the filter holder); plug the inlet to the first impinger (or filter holder); and pull a 380 mm (15 in.) Hg vacuum. If the leakage rate is >4% of the average sampling rate or if it is $\geq 0.00057 \text{ m}^3/\text{min}$ ($0.02 \text{ ft}^3/\text{m}$), whichever is less, it is unacceptable. This leak check is recommended but not mandatory.

3. Place a loosely packed filter of glass wool in the end of the probe if an external heated filter is not used, and connect the probe to the sampling train.

4. Attach a stack temperature sensor on the probe when required.

4.2.4 Sampling Train Operation (Constant Rate) - Sampling is performed at a constant rate of approximately $0.02 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{m}$) or less during the entire period as follows:

1. Record the initial dry gas meter readings, barometric pressures, and other data as indicated in Figure 4.2.
2. Position the tip of the probe at the first sampling point, and turn on the pump.
3. Adjust the sample flow to the predetermined constant rate of $0.021 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{m}$) or less.
4. Take other readings required by Figure 4.2 at least once at each sample point during each time increment.
5. Record the dry gas meter readings at the end of each time increment.
6. Record the stack gas temperature at each point when the stack gas is saturated or has water droplets.
7. The static pressure of the stack must also be determined when the moisture content is to be calculated using the partial pressure method.
8. Repeat steps 3 through 5 for each sampling point.
9. Turn off the pump, remove probe from the stack, and record the final readings after each traverse.
10. Leak check (as described in Subsection 4.2.3) after the last traverse, and record all leakage rates. This leak check is mandatory.
11. Cap the impingers with serum caps (or equivalent) and transport to the sample cleanup area if the train passes the leak check. If it does not, either reject the test results or correct the sample volume (see Section 3.4.6).
12. Check the sampling rate and the sample volume (ΔV_m) for each point. The volume for each point should be within $\pm 10\%$ of the average sample volume for all points. If all are within the limit, then the sample run is acceptable; otherwise, reject the results and either repeat the test run or consult the administrator.

4.3 Sample Recovery

Measure the volume of the condensed moisture to the nearest 1 ml. Determine the increase in weight of the silica gel (or gel plus impinger) to the nearest 0.5 g. Record these data on the data form shown in Figure 4.3 or on a similar form.

(45)

4.4 Sample Logistics, Data Collection, and Equipment Packing

Follow the above procedures until the required number of runs are completed. At the completion of the test:

1. Be sure that all data recorded during the field test are duplicated by using carbon paper or by using data forms and a laboratory notebook. Mail one set of data to the base laboratory, or give it to another team member or the agency, and have the other handcarried.

2. Examine all sampling equipment for damage and for proper packing for shipment. Label all shipping containers properly to prevent sample or equipment loss.

(414)

Table 4.1 ACTIVITY MATRIX FOR ON-SITE MEASUREMENT CHECKS

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|---|---|---|--|
| Condenser, addition of water and silica gel to system | 100 ml of distilled water in first two impingers; approximately 200 g of silica gel in fourth impinger | Either use graduated cylinder to measure water or weigh each impinger and its contents to nearest 0.5 g | Correct the additions |
| Assembling sampling train | <ol style="list-style-type: none"> 1. Assembled to specifications in Fig 1.1 2. Leak rate <4% or $0.00057 \text{ m}^3/\text{min}$ ($0.02 \text{ ft}^3/\text{min}$), whichever is less | <ol style="list-style-type: none"> 1. Assemble before each sample run 2. Leak check before sampling by plugging the nozzle or inlet to first impinger and pulling a vacuum of 380 mm (15 in.) Hg | <ol style="list-style-type: none"> 1. Reassemble 2. Correct leak |
| Sampling | <ol style="list-style-type: none"> 1. Sampling volume for each point within $\pm 10\%$ of average sample volume for all points 2. Minimum total sample gas volume of 0.60 sm^3 (21 sft^3) at a constant sampling rate $\leq 0.021 \text{ m}^3/\text{min}$ ($0.75 \text{ ft}^3/\text{min}$) 3. Minimum number and location of points specified by Method 1 | <ol style="list-style-type: none"> 1. Calculate for each test run 2. Make a quick calculation before testing; do an exact calculation after traverse 3. Check before the first test run by measuring duct and using Method 1 | <ol style="list-style-type: none"> 1. Repeat test run 2. As above 3. Repeat the procedure to comply with specifications of Method 1 |

(continued)

Table 4.1 (continued)

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|---|--|---|---|
| Sampling (cont.) | 4. Leakage rate <4% of the average sampling volume or <0.00057 m ³ /min (0.02 ft ³ /min), whichever is less | 4. Leak check after each test run or before equipment replacement during test at the maximum vacuum during the test (mandatory) | 4. Correct the sample volume, or repeat the sample run |
| Sample recovery | Volume of moisture condensed to nearest 1 ml; weight increase of silica gel to nearest 0.5 g | Use volumetric/gravimetric measurement | Repeat the measurement |
| Sample logistics, data collection, and packing of equipment | 1. All data recorded correctly

2. All equipment examined for damage and labeled for shipment

3. All sample containers and blanks properly labeled and packaged | 1. After completion of each test and before packing

2. As above

3. Visually check upon completion of each sample | 1. Complete the data

2. Repeat the sampling if damage occurred during test

3. Correct when possible |

5.0 POSTSAMPLING OPERATIONS

Table 5.1 at the end of this section summarizes the quality assurance activities for postsampling operations.

Posttest checks will have to be made on most of the sampling apparatus. The checks will include three calibration runs at a single orifice meter setting; cleaning; and/or routine maintenance. The cleaning and maintenance are discussed in APTD-0576². Figure 5.1 should be used to record the posttest checks.

5.1.1 Metering System - The metering system has two components that must be checked--the dry gas meter and the dry gas meter thermometer(s).

The dry gas meter thermometer(s) should be compared with the ASTM mercury-in-glass thermometer at room temperature. If the two readings agree within 6°C (10.8°F), they are acceptable; if not, the thermometer must be recalibrated according to Section 3.3.2 after the posttest check of the dry gas meter. For calculations, use the dry gas meter thermometer readings (field or recalibration values) that would give the higher temperatures--that is, if the field readings are higher, no correction is necessary, but if the recalibration value is higher, add the difference in the two readings to the average dry gas meter temperature reading.

The dry gas meter must be posttested (Section 3.3.2). The metering system should not have any of the leaks that were corrected prior to the posttest check. If the dry gas meter calibration factor (Y) does not deviate by >5% from the initial calibration factor, the dry gas meter volumes obtained during the test series are acceptable. If Y deviates by >5%, recalibrate the metering system (Section 3.3.2).

(457)

Date 1/2/79 Calibrated by 43P Blayman
Meter box number FM-1

Dry Gas Meter

Pretest calibration factor Y 0.986 (must be within $\pm 2\%$)*
Posttest checks, Y₁ 0.987 Y₂ _____ (must be within $\pm 5\%$ of pretest)
Recalibration required? _____ yes ☒ no
If yes, recalibration factor Y _____ (must be within $\pm 2\%$)*
Lower calibration factor, Y 0.936 for calculations (pretest or posttest)*

Dry Gas Thermometer

Was a pretest meter temperature correction used? _____ yes ☒ no
If yes, temperature correction _____ (within $\pm 3^\circ\text{C}$ (5.4°F) over range)*
Post test comparison with mercury-in-glass thermometer C.A.
(within $\pm 6^\circ\text{C}$ (10.8°F) at room temperature)
Recalibration required? _____ yes ☒ no
Recalibration temperature correction, if used _____ (within $\pm 3^\circ\text{C}$ (5.4°F) over range)*
If yes; no correction is necessary for calculations when meter thermometer temperature is higher.
If recalibration temperature is higher, add correction to average meter temperature for calculations

Barometer

Was pretest field barometer reading correct ☒ yes _____ no
Posttest comparison _____ mm (in.) Hg ± 2.5 mm (0.1 in.) Hg
Was recalibration required? _____ yes ☒ no
If yes; no correction is necessary for calculations when the field barometer has the lower reading
If the mercury-in-glass reading is lower, then subtract the difference from the field data readings for the calculation

Stack Gas Temperature Sensor (if required)

Average stack temperature 135°C (275°F)
Posttest comparison 135° [within $\pm 2^\circ\text{C}$ (4°F)]*
Was recalibration required? _____ yes ☒ no

*Most significant items/parameters to be checked.

Figure 5.1 Posttest equipment checks.

458

For the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.1.2 Barometer - The field barometer should be compared to the mercury-in-glass barometer. If the readings agree within ± 5 mm (0.2 in.) Hg, the field readings are acceptable; if not, use the lesser calibration value for the calculations. If the field barometer reads lower than the mercury-in-glass barometer, the field data are acceptable. If the mercury-in-glass barometer gives the lower reading, use the difference in the two readings (the adjusted barometric value) in the calculations.

5.1.3 Stack Gas Temperature Sensor - The stack gas temperature sensor should be compared with an ASTM mercury-in-glass reference thermometer. Place both the stack sensor and reference thermometer in an atmosphere (air or water) that is within $\pm 5^{\circ}\text{C}$ (10°F) of the average stack temperature. If both values agree within $\pm 2^{\circ}\text{C}$ (4°F) then the pretest calibration is acceptable. If not, then calculate the moisture content using both the pretest calibration and the posttest corrected values. If either or both calculated values are greater than the measured moisture, then either or both may be eliminated from any final emissions calculations.

(454)

Table 5.1 ACTIVITY MATRIX FOR POSTTEST OPERATIONS

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|---------------------------|---|---|---|
| Dry gas meter | Within $\pm 5\%$ of the initial calibration factor | Make three runs at a single, intermediate orifice setting and at highest vacuum occurring during test (Sec 3.3.2) | Recalibrate; use calibration factor that gives lesser sample volume |
| Dry gas meter thermometer | Within $\pm 6^{\circ}\text{C}$ (10.8°F) at room temperature | Compare with ASTM mercury-in-glass thermometer after each field test | Recalibrate; use higher temperature for calculations |
| Barometer | Within ± 5 mm (0.2 in.) Hg at ambient pressure | Compare with mercury-in-glass barometer after each field test | Recalibrate; use lower barometric values for calculations |
| Stack temperature sensor | Within $\pm 2^{\circ}\text{C}$ (4°F) of the reference check temperature | After each run, compare with reference temperature | Recalibrate; perform calculations with and without temperature correction |

(466)

6.0 CALCULATIONS

Calculation errors due to mathematical mistakes can be a large part of total system error. Therefore, each set of calculations should be repeated or spot-checked by a team member other than the one who performed them originally. If a difference greater than a typical roundoff error is detected, the calculations should be checked step by step until the source of error is found and corrected. A computer program can be advantageous in reducing calculation errors. If a standardized computer program is used, the original data entry should be checked; if differences are observed, a new computer run should be made. Table 6.2 at the end of this section summarizes the quality assurance activities for calculations.

Carryout calculations, retaining at least one significant digit beyond that of the acquired data. Roundoff after final calculations to two significant digits for each run or sample in accordance with the ASTM 380-76 procedures. Record the results on Figure 6.1A, 6.1B, or 6.1C.

6.1 Nomenclature

The terms defined and listed alphabetically herein are to be used in calculating dry gas and water vapor volumes and moisture contents, and in verifying constant sampling rate.

| | |
|---------------|--|
| B_{ws} | = Water vapor in the gas stream, proportion by volume |
| ΔH | = Average pressure differential across the orifice meter, mm (in.) H_2O |
| $\Delta H@_i$ | = Measurement of pressure differential across the orifice meter, mm (in.) H_2O |
| L_a | = Maximum acceptable leakage rate for either a pretest leak check or a leak check ₃ following a component change; equal to 0.00057 m ³ /min (0.01995 ft ³ /min) |
| M_w | = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole) |

| | |
|-----------------------|---|
| P_{bar} | = Barometric pressure, mm (in.) Hg |
| P_m | = Absolute pressure at the dry gas meter (for this method, same as barometric pressure), mm (in.) Hg |
| P_s | = Absolute stack pressure, mm (in.) Hg |
| P_{static} | = Static pressure of the stack, mm (in.) H_2O |
| P_{std} | = Standard absolute pressure, 760 mm (29.92 in.) Hg |
| ρ_w | = Density of water, 0.9982 g/ml (0.002201 lb/ml) |
| R | = Ideal gas constant, 0.06236 (mm Hg) (m^3)/(g-mole) (K) for metric units and 21.85 (in. Hg) (ft^3)/(lb-mole) ($^{\circ}R$) for English units |
| S.V.P. | = Saturated vapor pressure of water at average stack temperature, mm (in.) Hg |
| T_m | = Absolute average dry gas meter temperature, K ($^{\circ}R$) |
| T_{std} | = Standard absolute temperature, 298K (528 $^{\circ}R$) |
| V_f | = Final volume of condenser water, ml |
| V_i | = Initial volume of condenser water, ml |
| V_m | = Volume of gas sample measured by dry gas meter, dcm (dcf) |
| ΔV_m | = Incremental volume ₃ measured by dry gas meter at each traverse point, dm^3 (dcf) |
| $V_{m(\text{std})}$ | = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, $ds m^3$ (dscf) |
| v_s | = Stack gas velocity, calculated by Method 2, using data from Method 5, m/s (ft/s) |
| $V_{wc(\text{std})}$ | = Volume of condensed water vapor, corrected to standard conditions, sm^3 (scf) |
| $V_{wsg(\text{std})}$ | = Volume of water vapor collected in silica gel, corrected to standard conditions, sm^3 (scf) |
| W_f | = Final weight of silica gel or silica gel plus impinger, g |
| W_i | = Initial weight of silica gel or silica gel plus impinger, g |
| Y | = Dry gas meter calibration factor |

6.2 Condensed Water Vapor Volume

$$V_{wc(std)} = \frac{(V_f - V_i) \rho_w RT_{std}}{P_{std} M_w} = K_1 (V_f - V_i) \quad \text{Equation 6-1}$$

where

$$K_1 = 0.001333 \text{ m}^3/\text{ml} \text{ for metric units, or} \\ = 0.04707 \text{ ft}^3/\text{ml} \text{ for English units.}$$

6.3 Water Vapor Volume Collected in Silica Gel

$$V_{wsg(std)} = \frac{(W_f - W_i) RT_{std}}{P_{std} M_w} = K_2 (W_f - W_i) \quad \text{Equation 6-2}$$

where

$$K_2 = 0.001335 \text{ m}^3/\text{g} \text{ for metric units, or} \\ = 0.04715 \text{ ft}^3/\text{g} \text{ for English units.}$$

6.4 Dry Gas Volume, Corrected to Standard Conditions

Correct the sample volume measured by the dry gas meter to standard conditions (20°C and 760 mm Hg or 68°F and 29.92 in. Hg) by using Equation 6-3.

$$V_{m(std)} = V_m Y \frac{T_{std}}{T_m} \frac{P_m}{P_{std}} = \frac{K_3 V_m Y P_m}{T_m} \quad \text{Equation 6-3}$$

where

$$K_3 = 0.3858 \text{ K/mm Hg for metric units, or} \\ = 17.64 \text{ }^\circ\text{R/in. Hg for English units.}$$

Note: If the leak rate observed during any mandatory leak checks exceeds the specified acceptable rate (L_a), either the value of V_m in Equation 6-3 may be corrected (as described in Section 3.4.6 of Method 5) or the test run may be invalidated.

603

VOLUME OF WATER VAPOR CONDENSED IN IMPINGERS

$$V_f = 271 \text{ ml}, V_i = 200 \text{ ml}$$

$$V_{wc(std)} = 0.04707 (V_f - V_i) = 0.332 \text{ ft}^3 \quad \text{Equation 6-1}$$

VOLUME OF WATER VAPOR COLLECTED IN SILICA GEL

$$W_f = 215 \text{ g}, W_i = 205 \text{ g}$$

$$V_{wsg(std)} = 0.04715 (W_f - W_i) = 0.542 \text{ ft}^3 \quad \text{Equation 6-2}$$

SAMPLE VOLUME

$$V_m = 31.54 \text{ ft}^3, T_m = 53.2^\circ \text{R}, P_m = 29.00 \text{ in. Hg}$$

$$Y = 1.016$$

$$V_{m(std)} = 17.64 \frac{V_m Y P_m}{T_m} = 30.425 \text{ ft}^3 \quad \text{Equation 6-3}$$

MOISTURE CONTENT

$$V_{wc(std)} = 0.332 \text{ ft}^3, V_{wsg(std)} = 0.542 \text{ ft}^3,$$

$$V_{m(std)} = 30.425 \text{ ft}^3$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} = 0.113 \quad \text{Equation 6-4}$$

Figure 6.1A Moisture content calculation form (English units).

VOLUME OF WATER VAPOR CONDENSED IN IMPINGERS

$$V_f = 271 \text{ ml}, V_i = 200 \text{ ml}$$

$$V_{wc(std)} = 0.001333 (V_f - V_i) = 0.0914 \text{ m}^3 \quad \text{Equation 6-1}$$

VOLUME OF WATER VAPOR COLLECTED IN SILICA GEL

$$W_f = 215.0 \text{ g}, W_i = 203.5 \text{ g}$$

$$V_{wsg(std)} = 0.001335 (W_f - W_i) = 0.0154 \text{ m}^3 \quad \text{Equation 6-2}$$

SAMPLE VOLUME

$$V_m = 0.884 \text{ m}^3, T_m = 299.0 \text{ }^\circ\text{K}, P_m = 736.6 \text{ mm Hg}$$

$$Y = 1.014$$

$$V_{m(std)} = 0.3858 \frac{V_m Y P_m}{T_m} = 0.8569 \text{ m}^3 \quad \text{Equation 6-3}$$

MOISTURE CONTENT

$$V_{wc(std)} = 0.0914 \text{ m}^3, V_{wsg(std)} = 0.0154 \text{ m}^3,$$

$$V_{m(std)} = 0.8569 \text{ m}^3$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} = 0.114 \quad \text{Equation 6-4}$$

Figure 6.1B. Moisture content calculation form (metric units).

(465)

MOISTURE CONTENT

$$t_s \text{ avg} = 121.6. ^\circ\text{F}, 49.83 ^\circ\text{C}$$

$$P_{\text{bar}} = 29.00 \text{ in. Hg}, 736.6 \text{ mm Hg}$$

$$P_{\text{static}} = -1.50 \text{ in. H}_2\text{O}, -3.81 \text{ mm H}_2\text{O}$$

$$\text{S.V.P.} = 3.60 \text{ in. Hg}, 92.0 \text{ mm Hg}$$

$$B_{\text{ws}} = \frac{\text{S.V.P.}}{P_{\text{bar}} + \frac{P_{\text{static}}}{13.6}} = 0.125$$

Figure 6.1C Moisture content calculation form using saturation vapor pressure (English and metric units).

6.5 Moisture Content

$$B_{\text{ws}} = \frac{V_{\text{wc(std)}} + V_{\text{wsg(std)}}}{V_{\text{wc(std)}} + V_{\text{wsg(std)}} + V_{\text{m(std)}}} \quad \text{Equation 6-4}$$

Note: In moisture saturated or droplet-laden gas streams, two calculations of the moisture content of the stack gas should be made--one using a value based on the saturated conditions (Equation 6-5) and another using the results of the impinger analysis. The lower of these two B_{ws} values should be considered correct.

To determine the moisture content in moisture saturated or droplet-laden gas streams, attach a temperature sensor capable of measuring $\pm 1^\circ\text{C}$ (2°F) to the probe; measure the stack gas temperature at each traverse point during the traverse; measure the absolute stack pressure. Determine the moisture percentage, either by:

466

1. Using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or

2. Using saturation vapor pressure Tables 6.1A and 6.1B and Equation 6-5.

$$B_{ws} = \frac{S.V.P.}{P_{bar} + \frac{P_{static}}{13.6}} \quad \text{Equation 6-5}$$

If the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods approved by the administrator should be used.

6.6 Constant Sampling Rate Verification

For each sample point, determine the ΔV_m and calculate the average. If the value for any sample point differs from the average by >10%, reject the results and repeat the run.

Table 6.1A VAPOR PRESSURE OF WATER AT SATURATION (°F), in. Hg

| Temp
°F | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 50 | .3626 | .3764 | .3906 | .4052 | .4203 | .4359 | .4520 | .4586 | .4858 | .5035 |
| 60 | .5218 | .5407 | .5601 | .5802 | .6009 | .6222 | .6442 | .6669 | .6903 | .7144 |
| 70 | .7392 | .7648 | .7912 | .8183 | .8462 | .8750 | .9046 | .9352 | .9666 | .9989 |
| 80 | 1.032 | 1.066 | 1.102 | 1.138 | 1.175 | 1.213 | 1.253 | 1.293 | 1.335 | 1.378 |
| 90 | 1.422 | 1.467 | 1.513 | 1.561 | 1.610 | 1.660 | 1.712 | 1.765 | 1.819 | 1.875 |
| 100 | 1.932 | 1.992 | 2.052 | 2.114 | 2.178 | 2.243 | 2.310 | 2.379 | 2.449 | 2.921 |
| 110 | 2.596 | 2.672 | 2.749 | 2.829 | 2.911 | 2.995 | 3.081 | 3.169 | 3.259 | 3.351 |
| 120 | 3.446 | 3.543 | 3.642 | 3.744 | 3.848 | 3.954 | 4.063 | 4.174 | 4.289 | 4.406 |
| 130 | 4.525 | 4.647 | 4.772 | 4.900 | 5.031 | 5.165 | 5.302 | 5.442 | 5.585 | 5.732 |
| 140 | 5.881 | 6.034 | 6.190 | 6.380 | 6.513 | 6.680 | 6.850 | 7.024 | 7.202 | 7.384 |
| 150 | 7.569 | 7.759 | 7.952 | 8.150 | 8.351 | 8.557 | 8.767 | 8.981 | 9.200 | 9.424 |
| 160 | 9.652 | 9.885 | 10.12 | 10.36 | 10.61 | 10.86 | 11.12 | 11.38 | 11.65 | 11.92 |
| 170 | 12.20 | 12.48 | 12.77 | 13.07 | 13.37 | 13.67 | 13.98 | 14.30 | 14.62 | 14.96 |
| 180 | 15.29 | 15.63 | 15.98 | 16.34 | 16.70 | 17.07 | 17.44 | 17.82 | 18.21 | 18.61 |

(467)

Table 6.1B VAPOR PRESSURE OF WATER AT SATURATION (°C), mm Hg

| Temp
°C | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| 10 | 9.20 | 9.92 | 10.67 | 11.07 | 11.65 | 12.79 | 13.73 | 14.74 | 15.26 | 16.36 |
| 20 | 17.50 | 18.77 | 20.1 | 20.78 | 22.23 | 23.80 | 25.15 | 27.09 | 27.98 | 29.85 |
| 30 | 31.83 | 33.91 | 36.12 | 37.26 | 39.65 | 42.16 | 44.83 | 47.63 | 49.07 | 52.12 |
| 40 | 55.32 | 58.67 | 62.20 | 64.03 | 67.87 | 71.86 | 76.07 | 80.49 | 82.78 | 87.53 |
| 50 | 92.51 | 97.74 | 103.20 | 106.02 | 111.91 | 118.03 | 124.46 | 131.19 | 134.67 | 141.86 |
| 60 | 149.38 | 157.23 | 165.43 | 169.67 | 178.41 | 187.55 | 197.08 | 207.01 | 212.12 | 222.68 |
| 70 | 233.68 | 245.16 | 257.05 | 263.14 | 275.84 | 289.05 | 302.77 | 316.99 | 324.36 | 339.60 |
| 80 | 355.09 | 371.35 | 388.37 | | | | | | | |

Table 6.2 ACTIVITY MATRIX FOR CALCULATION CHECKS

| Characteristics | Acceptance limits | Frequency and method of measurement | Action if requirements are not met |
|----------------------|---|---|--|
| Analytical data form | All data and calculations are shown | Visual check | Complete missing data values |
| Calculations | Difference between check and original calculations should not exceed roundoff error | Repeat all calculations starting with raw data for hand calculations; check all raw data input for computer calculations and hand calculate one sample per test | Indicate errors on analytical data form, Fig 4.2 |

468

7.0 MAINTENANCE

Normal use of emission testing equipment subjects it to corrosive gases, temperature extremes, vibrations, and shocks. Keeping the equipment in good operating order over an extended period of time requires routine maintenance and knowledge of the equipment. Maintenance of the entire sampling train should be performed either quarterly or after 1000 ft³ of operation, whichever occurs sooner. Maintenance procedures are summarized in Table 7.1 at the end of this section. The following procedures are recommended, but not required, to increase the reliability of the equipment.

7.1 Pumps

Several types of pumps are used in commercial sampling trains. Two of the most common types are the fiber vane pump with in-line oiler and the diaphragm pump. The fiber vane pump requires a periodic check of the oil and the oiler jar. The used oil (usually low nondetergent or machine weight) should be about the same translucent color as the unused or spare oil. When the fiber vane pump starts to run erratically or when the head is removed each year, the fiber vanes should be changed.

The diaphragm pump requires little maintenance. If the diaphragm pump leaks or runs erratically, it is normally due to a bad diaphragm or malfunctions in the valves; these parts are easily replaced and should be cleaned annually by complete disassembly of the train.

7.2 Dry Gas Meters

The dry gas meter should be checked for excess oil and component corrosion by removing the top plate every 3 mo. The meter should be disassembled and all components cleaned and checked more often if the dials show erratic rotation, or if the meter will not calibrate properly.

7.3 Inclined Manometer

The fluid should be changed when it is discolored or contains visible matter and when it is disassembled yearly. No

(c/c-6)

other routine maintenance is required since the inclined manometer is checked during the leak checks of both the pitot tube and the entire meter box.

7.4 Sampling Train

All remaining sample train components should be visually checked every 3 mo, and they should be completely disassembled and cleaned or replaced yearly. Many of the items, such as quick disconnects, should be replaced when damaged rather than after they are periodically checked. Normally, the best maintenance procedure is to replace the entire unit--for example, a meter box, sample box, or umbilical cord.

4/16

Table 7.1 ACTIVITY MATRIX FOR EQUIPMENT MAINTENANCE CHECKS

| Apparatus | Acceptance limits | Frequency and method of measurements | Action if requirements are not met |
|----------------------|---|--|---|
| Routine main-tenance | No erratic behavior | Routine maintenance quarterly; disassemble and clean yearly | Replace parts as needed |
| Fiber vane pump | Leak free and required flow | Periodic check of oil jar; remove head, and change fiber vanes | Replace as needed |
| Diaphragm pump | Leak free valves functioning properly with required flow | Clean valves during yearly disassembly | Replace when leaking or when running erratically |
| Dry gas meter | No excess oil, corrosion, or erratic rotation of the dial | Check every 3 mo for excess oil or corrosion by removing top plate; check valves and diaphragm when meter dial runs erratically or when meter will not calibrate | Replace parts as needed, or replace meter |
| Inclined manometer | No discoloration or visible matter in the fluid | Check periodically; change fluid during yearly disassembly | Replace parts as needed |
| Sampling train | No damage | Visually check every 3 mo; completely disassemble and clean or replace yearly | If failure noted, use another entire control console, sample box, or umbilical cord |

8.0 AUDITING PROCEDURE

An audit is an independent assessment of data quality. Independence is achieved by using apparatus and standards that are different from those used by the regular field crew. Routine quality assurance checks by a field team are necessary for obtaining good quality data, but they are not part of the auditing procedure. Table 8.1 at the end of this section summarizes the quality assurance activities for the auditing.

Based on the results of the collaborative tests of Method 4, a performance audit of data processing and a systems audit are recommended. These two audits are described in the Subsections 8.1 and 8.2.

8.1 Performance Audit of Data Processing

Performance audits are conducted by the auditor to quantitatively evaluate the quality of the data produced by the total measurement system (sample collection, sample analysis, and data processing). Due to the limited sizes of most emission-testing companies, it is recommended that these audits be performed by the responsible control agency once during every enforcement source test, regardless of whether the tests are conducted by agency or private company personnel. A source test for enforcement comprises a series of runs at one source.

Calculation errors are prevalent in Method 4. Data processing errors can be determined by auditing the data recorded on the field and the laboratory forms. The original and the check calculations should agree. If not, all of the data and calculations should be checked. The calculation errors should be clearly explained to the source-test team to prevent or minimize reoccurrence. The data processing errors may also be determined by requesting that copies of data sets compiled in the field and copies of manual data reductions (or computer printouts if used) be forwarded to the evaluator for audit.

8.2 Systems Audit

A systems audit is an on-site qualitative inspection and review of the quality assurance method used by the test team for

the total measurement system (sample collection, sample analysis, data processing, etc.). Initially, a systems audit specified by a quality assurance coordinator should be conducted for each enforcement source test, which by definition comprises three runs at one source. After the team gains experience with the method, the frequency of audit may be reduced--for example, once for every four tests.

The functions of the auditor are summarized by the following:

1. Observe procedures and techniques of the field team during sample collection.
2. Check/verify the records of apparatus calibration.
3. Record the results of the audit and forward them with comments on source team management to the quality assurance coordinator so that any needed corrective actions may be implemented.

The auditor should observe the field team's overall performance of the source test. Specific operations to observe should include (but not be limited to):

1. Setting up and leak testing the sampling train.
2. Constant rate sampling check of the sampling train.
3. Final leak check of train.
4. Sample recovery.

Figure 8.1 is a suggested checklist to be used by the auditor for developing a list of important techniques/steps to observe.

474

| Yes | No | Operation |
|----------|-------|---|
| | | <u>Presampling preparation</u> |
| _____ | _____ | 1. Knowledge of process conditions |
| _____ | _____ | 2. Calibration of pertinent equipment prior to each field test; in particular, the dry gas meter should be checked before each test |
| | | <u>On-site measurements</u> |
| _____ | _____ | 3. Leak testing of sample train after sample run |
| _____ | _____ | 4. Addition of water and silica gel to impingers, and correct location of impingers |
| _____ | _____ | 5. Constant sampling rate and not exceeding specified rate |
| _____ | _____ | 6. Measurement of condensed water to within specified limits |
| _____ | _____ | 7. Record of pertinent process condition during sample collection |
| _____ | _____ | 8. Probe maintained at given temperature |
| | | <u>Postsampling</u> |
| _____ | _____ | 9. Calculation procedure/check |
| _____ | _____ | 10. Calibration checks |
| COMMENTS | | |
| | | |

Figure 8.1 Method 4 checklist to be used by auditors.

Table 8.1 ACTIVITY MATRIX FOR AUDITING PROCEDURES

| Audit | Acceptance limits | Frequency of method of measurement | Action if requirements are not met |
|--|---|--|--|
| Data processing errors | Original and check calculations should agree | Once during every enforcement source test, do independent calculations starting with recorded data | Check and correct all data for the source test |
| Systems audit--observance of technique | Operation/technique described in this section of the Handbook | Once during every enforcement test until experience gained, then every fourth test; observe techniques; use audit checklist, Fig 8.1 | Explain to team the deviations from recommended techniques; note the deviations on Fig 8.1 |

(474)

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two considerations are necessary: (1) the measurement process must be in a state of statistical control, and (2) the systematic errors, when combined with the random variations (errors of measurement), must result in an acceptable level of uncertainty. To ensure good data, it is necessary to perform quality control checks and independent audits of the measurement process; and to use materials, instruments, and procedures which can be traced to a standard of reference.

The working calibration standards should be traceable to primary or higher level standards such as those for the dry gas meter. The dry gas meter should be calibrated against a wet test meter which has been verified by an independent liquid displacement meter.

(67)

10.0 REFERENCE METHOD



METHOD 4—DETERMINATION OF MOISTURE IN STACK GASES

1. Principle and Applicability.

1.1 Principle. A gas sample is extracted proportionally from the source and moisture is removed from the gas stream, condensed, and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for the determination of moisture in stack gas.

Two methods are given. One is a reference method for the accurate determination of moisture content as needed to calculate emission data. The other is an approximation method for moisture content to be subsequently used for setting isokinetic sampling rates. For this latter purpose, the tester may use any alternate means for approximating the moisture content, e.g. drying tubes, wet bulb-dry bulb technique, condensation techniques, stoichiometric calculations, previous experience, etc. However, the actual isokinetic rate maintained during a pollutant sampling run and the moisture content used to calculate emission data will not be based on the results of the approximation method (see exception in note below), but will be determined from the data of the reference method, which is normally conducted simultaneously with a pollutant measurement run.

Note.—Any of the approximation methods which are shown to the satisfaction of the Administration of yielding results to within 1% H₂O of the reference method results may be used in lieu of the reference method.

These methods are not applicable to gas streams that contain liquid droplets. For these cases, assume that the gas stream is saturated. Determine the average stack gas temperature using gauges described in Method 2 and by traversing according to Method 1. Then obtain the moisture percentage by (1) using a psychometric chart and making appropriate corrections, if stack pressure is different from that of the chart,

for absolute pressure or (2) by using saturation vapor pressure tables.

2. Reference Method.

The procedure for determining moisture content described in Method 5 is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

2.1.1 Probe—Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter.

2.1.2 Condenser—Any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law or (2) by passing the sample gas stream through a tared silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

2.1.3 Cooling system—Ice bath container and crushed ice, or equivalent, to aid in condensing moisture.

2.1.4 Drying tube—Tube packed with 6-16 mesh indicating-type silica gel, or equivalent, to dry the sample gas and protect the pump and dry gas meter. This may be an integral part of the condenser system, in which case the tube shall be immersed in the ice bath and a thermometer placed at the outlet for monitoring purposes. If approach (1) of section 2.1.2 is used to measure the moisture leaving the condenser, the temperature and pressure must be monitored before the silica gel tube.

2.1.5 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter with ± 2 percent accuracy, and related equipment, or other metering systems approved by the Administrator, as required to maintain a proportional sampling rate and to determine sample gas volume.

2.1.6 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.



25

Figure 4-2. Field moisture determination-reference method.

| | IMPINGER
VOLUME,
ml | SILICA GEL
WEIGHT,
g |
|------------|---------------------------|----------------------------|
| FINAL | | |
| INITIAL | | |
| DIFFERENCE | | |

Figure 4-3. Analytical data-reference method.

2.1.7 Pitot tube—Type S, or equivalent, attached to probe to allow constant monitoring of the stack gas velocity so that the sampling flow rate can be regulated proportional to the stack gas velocity. The tips of the probe and pitot tube shall be adjacent to each other and the free space between them shall be about 1.9 cm (0.75 in.). When used with this method, the pitot tube need not be calibrated.

2.1.8 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value or ± 0.013 mm (0.0005 in.), in whichever is greater. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with the pulsating flow. Thus, methods or other devices acceptable to the Administrator may be used when conditions warrant.

2.1.9 Temperature gauge—Thermocouple, liquid filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauges that are capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature.

2.1.10 Graduated cylinder and/or balance—To measure condensed water and moisture caught in the silica gel to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

2.1.11 Temperature and pressure gauges—If Dalton's law is used to monitor temperature and pressure at condenser outlet. The temperature gauge shall have an accuracy of 1°C (2°F). The pressure gauge shall be capable of measuring pressure to within 2.5 mm Hg (0.1 in. Hg).

2.1.12 Silica gel—If used to measure moisture leaving condenser, indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

2.2 Procedure. The procedure below is written for a condenser system incorporating silica gel and gravimetric analysis to measure the moisture leaving the condenser and volumetric analysis to measure the condensed moisture.

2.2.1 Select the sampling site and minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the range of velocity head using Method 2 for the purpose of making proportional sampling rate calculations.

Select a suitable velocity head to correspond to about $0.014\text{ m}^3/\text{min}$ (0.5 cfm). Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides (four total sampling ports) for large stacks to enable use of shorter probe lengths. Mark probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Weigh and record weight of silica gel to the nearest 0.5 g.

2.2.2 Select a suitable total sampling time of no less than 1 hour such that a minimum total gas sample volume of 0.6 m^3 (20 ft³) at standard conditions will be collected and the sampling time per traverse point is not less than 2 min., or some greater time interval as specified by the Administrator.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heating system to about 120°C (248°F) so as to prevent water condensation and allow time for temperature to stabilize. Place crushed ice in the ice bath container. Leak check the train by plugging the probe inlet and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4 percent of the average sampling rate or $0.00057\text{ m}^3/\text{min}$ (0.02 cfm), which ever is less, is unacceptable.

2.2.4 During the sampling run, maintain a sampling rate within 20 percent, or as specified by the Administrator, of constant proportionality. For each run, record the data required on the example data sheet shown in Figure 4-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter reading at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at each sample point at least once during each time increment.

2.2.5 To begin sampling position the probe tip at the first traverse point. Immediately start the pump and adjust the flow to proportional conditions. Traverse the cross section. Add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the silica gel outlet to avoid excessive moisture losses.

2.2.6 After collecting the sample, measure the volume increase of the liquid to the nearest 1 ml. Determine the increase in weight of the silica gel tube to the nearest 0.5 g. Record the information (see example, data sheet, Figure 4-3) and calculate the moisture percentage.

2.3 Calculations. Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

2.3.1 Nomenclature.

B_w = Proportion by volume
 M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)
 P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg)
 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)
 R = Ideal gas constant, 0.06236 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.83 (in. Hg)(ft³)/(lb-mole)(°R) for English units
 T_m = Absolute temperature at meter, °K (°R)
 T_{std} = Absolute temperature, 293° K (528° R)
 V_m = Dry gas volume measured by meter, dcm (dcf)
 $V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf)
 $V_{ws(std)}$ = Volume of water vapor condensed corrected to standard conditions, m³ (ft³)
 $V_{wsq(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, m³ (ft³)
 V_f = Final volume of condenser contents, ml
 V_i = Initial volume, if any, of condenser contents, ml
 W_f = Final weight of condenser contents, g
 W_i = Initial weight of condenser contents, g
 ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)

2.3.2 Volume of water vapor condensed.

$$V_{ws(std)} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} = K \frac{V_m P_m}{T_m} \quad \text{Equation 4-3}$$

where:
 $K = 0.3855 \text{ } ^\circ\text{K/mm Hg for metric units}$
 $= 17.65 \text{ } ^\circ\text{R/in. Hg for English units}$

2.3.5 Moisture Content:

$$B_w = \frac{V_{ws} + V_{wsq}}{V_{ws} + V_{wsq} + V_{m}}$$

Equation 4-4

where:

$K = 0.00134 \text{ m}^3/\text{ml for metric units}$

$= 0.0472 \text{ ft}^3/\text{ml for English units}$

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsq(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K (W_f - W_i)$$

Equation 4-2

where:

$K = 0.00134 \text{ m}^3/\text{g for metric units}$

$= 0.0472 \text{ ft}^3/\text{g for English units}$

2.3.4 Gas volume.

$$V_{m(std)} = V_m \frac{(P_m)(T_{std})}{(P_{std})(T_m)}$$

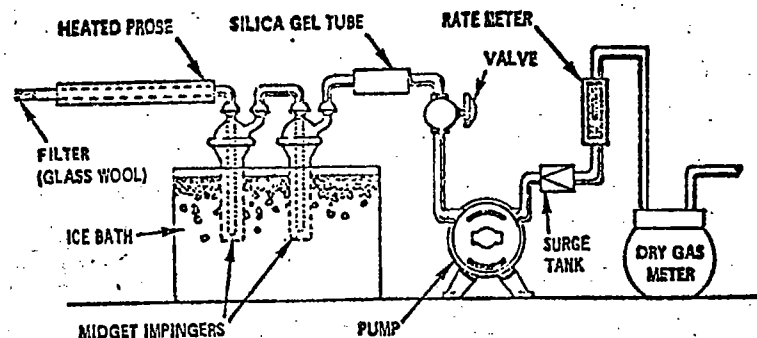


Figure 4-4. Moisture-sampling train.

LOCATION _____ COMMENTS _____
 TEST _____
 DATE _____
 OPERATOR _____
 BAROMETRIC PRESSURE _____

| CLOCK TIME | GAS VOLUME THROUGH METER, (Vm), m ³ (ft ³) | RATE METER SETTING m ³ /min. (ft ³ /min.) | METER TEMPERATURE, °C (°F) |
|------------|---|---|----------------------------|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

Figure 4-5. Field moisture determination.

Section No. 3.3.10
 Revision No. 0
 Date January 15, 1980
 Page 4 of 5

3.1.3 Ice bath—Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying tube—Tube packed with 8-16 mesh indicating-type silica gel, or equivalent, to dry the sample gas and to protect the meter and pump.

3.1.5 Valve—Needle valve, to regulate sample gas flow rate.

3.1.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through the train.

3.1.7 Volume meter—Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, and calibrated over the range of flow rates and conditions actually used during sampling.

3.1.8 Rate meter—Rotameter, to measure the flow range from 0 to 3 lpm (0 to 0.11 cfm).

3.1.9 Graduated cylinder—25 ml.

3.1.10 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decreases.

3.1.11 Vacuum gauge—At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-4. Leak check by placing a vacuum gauge at the inlet to the first impinger and drawing a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet of the rotameter, and then turning off the pump. The vacuum shall remain constant for a least one minute. Carefully release the vacuum gauge before releasing the rotameter end.

3.2.2 Connect the probe and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-5.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas and therefore other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

B_{ws} = Approximate water vapor in the gas stream leaving the impinger, 0.025 proportion by volume

B_{ws} = Water vapor in the gas stream, proportion by volume

M_w = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole)

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg)

P = Ideal gas constant, 0.08206 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.83 (in. Hg)(ft³)/(lb-mole)(°R) for English units

T_m = Absolute temperature at meter, °K (°R)

T_{std} = Standard absolute temperature, 293° K (528° R)

V_f = Final volume of impinger contents, ml

V_i = Initial volume of impinger contents, ml

V_m = Dry gas volume measured by dry gas meter, dscm (dscf)

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf)

$V_{ws(std)}$ = Volume of water vapor condensed, corrected to standard conditions, m³ (ft³)

ρ_w = Density of water, 1 g/ml (0.00220 lb/ml)

3.3.2 Volume of water vapor collected.

$$V_{ws} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w}$$

$$B_{ws} = \frac{V_{ws}}{V_{ws} + V_{m(std)}} + B_{wm} = \frac{V_{ws}}{V_{ws} + V_{m(std)}} + (0.025) \quad \text{Equation 4-7}$$

4. Calibration.

4.1 Use methods and equipment as specified in Methods 2 and 5 and APTD-0576 to calibrate dry gas meter, barometer, and thermometers.

5. References.

5.1 Air Pollution Engineering Manual, Danielson, J. A. (ed.), U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999-AP-40, 1967.

5.2 Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

5.3 Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

$$= K(V_f - V_i) \quad \text{Equation 4-5}$$

Where:

$K = 0.00134 \text{ m}^3/\text{ml}$ for metric units

$= 0.0472 \text{ ft}^3/\text{ml}$ for English units

3.3.3 Gas volume.

$$V_{m(std)} = V_m \left(\frac{P_m}{P_{std}} \right) \left(\frac{T_{std}}{T_m} \right)$$

$$= K \frac{V_m P_m}{T_m}$$

where:

$K = 0.3855 \text{ }^\circ\text{K}/\text{mm Hg}$ for metric units

$= 17.65 \text{ }^\circ\text{R}/\text{in. Hg}$ for English units

3.3.4 Approximate moisture content.

11.0 REFERENCES

1. Martin, R. W. Construction Details of Isokinetic Source Sampling Equipment. APTD-0581. Air Pollution Control Office, EPA, Research Triangle Park, N.C., 1971.
2. Rom, J. J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. APTD-0576. Office of Air Programs, EPA, Research Triangle Park, N.C., 1972.
3. Midgett, M. R. The EPA Program for the Standardization of Stationary Source Emission Test Methodology--A Review. EPA-600/4-76-044. Environmental Monitoring and Support Laboratory, EPA, Research Triangle Park, N.C., 1976.

12.0 DATA FORMS

Blank data forms are provided on the following pages for the convenience of the Handbook user. Each blank form has the customary descriptive title centered at the top of the page. However, the section-page documentation in the top right-hand corner of each page of other sections has been replaced with a number in the lower right-hand corner that will enable the user to identify and refer to a similar filled-in form in a text section. For example, Form M4-1.2 indicates that the form is Figure 1.2 in Section 3.3.1 of the Method 4 Handbook. Future revisions of these forms, if any, can be documented by 1.2A, 1.2B, etc. Eight of the blank forms listed below are included in this section. Four are in the Method Highlights Section, as shown by the MH following the form number.

| <u>Form</u> | <u>Title</u> |
|-------------|--|
| 1.2 | Procurement Log |
| 2.3A & B | Meter Box Calibration Data and Calculation Form (English and Metric units) |
| 2.4A & B | Posttest Meter Calibration Data Form (English and Metric units) |
| 2.5 (MH) | Pretest Sampling Checks |
| 3.1 (MH) | Pretest Preparation Checklist |
| 4.1 (MH) | On-Site Measurement Checklist |
| 4.2 | Method 4 Field and Sample Recovery Data Form |
| 4.3 | Method 4 Analytical Data Form |
| 5.1 (MH) | Posttest Equipment Checks |
| 6.1A & B | Moisture Content Calculation Form (English and Metric units) |
| 6.1.C | Moisture Content Calculation Form (English and Metric units) |
| 8.1 | Method 4 Checklist To Be Used By Auditors |

487

[illegible]

METER BOX CALIBRATION DATA AND CALCULATION FORM

(English units)

Date _____

Meter box number _____

Barometric pressure, P_b = _____ in. Hg Calibrated by _____

| Orifice
manometer
setting
(ΔH),
in. H ₂ O | Gas volume | | Temperature | | | | Time
(θ),
min | Y_i | $\Delta H@_i$
in. H ₂ O |
|--|--|---|---------------------------------------|-------------------------------|--------------------------------|--------------------------------------|------------------------------|-------|---------------------------------------|
| | Wet test
meter
(V_w),
ft ³ | Dry gas
meter
(V_d),
ft ³ | Wet test
meter
(t_w),
°F | Dry gas meter | | | | | |
| | | | | Inlet
(t_{d_i}),
°F | Outlet
(t_{d_o}),
°F | Avg ^a
(t_d),
°F | | | |
| 0.5 | 5 | | | | | | | | |
| 1.0 | 5 | | | | | | | | |
| 1.5 | 10 | | | | | | | | |
| 2.0 | 10 | | | | | | | | |
| 3.0 | 10 | | | | | | | | |
| 4.0 | 10 | | | | | | | | |
| Avg | | | | | | | | | |

| ΔH ,
in.
H_2O | $\frac{\Delta H}{13.6}$ | $Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t + 460)}$ | $\Delta H@_i = \frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460) \theta}{V_w} \right]^2$ |
|-------------------------------|-------------------------|---|---|
| 0.5 | 0.0368 | | |
| 1.0 | 0.0737 | | |
| 1.5 | 0.110 | | |
| 2.0 | 0.147 | | |
| 3.0 | 0.221 | | |
| 4.0 | 0.294 | | |

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d .

(1/8/4)

METER BOX CALIBRATION DATA AND CALCULATION FORM (English units)

Nomenclature:

V_w = Gas volume passing through the wet test meter, ft^3 .

V_d = Gas volume passing through the dry gas meter, ft^3 .

t_w = Temperature of the gas in the wet test meter, $^{\circ}\text{F}$.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, $^{\circ}\text{F}$.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, $^{\circ}\text{F}$.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average t_{d_i} and t_{d_o} , $^{\circ}\text{F}$.

ΔH = Pressure differential across orifice, in. H_2O .

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run; tolerance $Y_i = Y \pm 0.02Y$.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all six runs; tolerance $Y = Y \pm 0.01Y$.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives $0.75 \text{ ft}^3/\text{min}$ of air at standard conditions for each calibration run, in. H_2O ; tolerance = $\Delta H@ \pm 0.15$ (recommended).

$\Delta H@$ = Average orifice pressure differential that gives $0.75 \text{ ft}^3/\text{min}$ of air at standard conditions for all six runs, in. H_2O ; tolerance = 1.84 ± 0.25 (recommended).

θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

447

METER BOX CALIBRATION DATA AND CALCULATION FORM

(Metric units)

Date _____

Meter box number _____

Barometric pressure, $P_b =$ _____ mm Hg Calibrated by _____

| Orifice
manometer
setting
(ΔH),
mm H ₂ O | Gas volume | | Temperature | | | | Time
(Θ),
min | Y _i | $\Delta H@_i$,
mm H ₂ O |
|---|---|--|---|---|--|--|------------------------------|----------------|--|
| | Wet test
meter
(V _w),
m ³ | Dry gas
meter
(V _d),
m ³ | Wet test
meter
(t _w),
°C | Dry gas meter | | | | | |
| | | | | Inlet
(t _{d_i}),
°C | Outlet
(t _{d_o}),
°C | Avg ^a
(t _d),
°C | | | |
| 10 | 0.15 | | | | | | | | |
| 25 | 0.15 | | | | | | | | |
| 40 | 0.30 | | | | | | | | |
| 50 | 0.30 | | | | | | | | |
| 75 | 0.30 | | | | | | | | |
| 100 | 0.30 | | | | | | | | |
| Avg | | | | | | | | | |

| ΔH ,
mm
H_2O | $\frac{\Delta H}{13.6}$ | $Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_d + \frac{\Delta H}{13.6}) (t_w + 273)}$ | $\Delta H@_i = \frac{0.00117 \Delta H}{P_b (t_d + 273)} \left[\frac{(t_w + 273) \Theta}{V_w} \right]^2$ |
|------------------------------|-------------------------|---|--|
| 10 | 0.7 | | |
| 25 | 1.8 | | |
| 40 | 2.94 | | |
| 50 | 3.68 | | |
| 75 | 5.51 | | |
| 100 | 7.35 | | |

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d .

METER BOX CALIBRATION DATA AND CALCULATION FORM (metric units)

Nomenclature:

V_w = Gas volume passing through the wet test meter, m^3 .

V_d = Gas volume passing through the dry gas meter, m^3 .

t_w = Temperature of the gas in the wet test meter, $^{\circ}C$.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, $^{\circ}C$.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, $^{\circ}C$.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o} , $^{\circ}C$.

ΔH = Pressure differential across orifice, mm H_2O .

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run; tolerance $Y_i = Y \pm 0.02Y$.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all six runs; tolerance $Y = Y \pm 0.01Y$.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives $0.021 m^3$ of air at standard conditions for each calibration run, mm H_2O ; tolerance $\Delta H@_i = \Delta H@ \pm 3.8$ mm H_2O (recommended).

$\Delta H@$ = Average orifice pressure differential that gives $0.021 m^3$ of air at standard conditions for all six runs, mm H_2O ; tolerance $\Delta H@ = 46.74 \pm 6.3$ mm H_2O (recommended).

θ = Time of each calibration run, min.

P_b = Barometric pressure, mm Hg.

POSTTEST DRY GAS METER CALIBRATION DATA FORM (English units)

Test number _____ Date _____ Meter box number _____ Plant _____

Barometric pressure, P_b = _____ in. Hg Dry gas meter number _____ Pretest Y _____

| Orifice
manometer
setting,
(ΔH),
in. H ₂ O | Gas volume | | Temperature | | | | Time
(Θ),
min | Vacuum
setting,
in. Hg | Y _i | $\frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6} \right) (t_w + 460)}$ |
|---|--|---|---|---|--|--|---------------------|------------------------------|----------------|--|
| | Wet test
meter
(V _w),
ft ³ | Dry gas
meter
(V _d),
ft ³ | Wet test
meter
(t _w),
°F | Dry gas meter | | | | | | |
| | | | | Inlet
(t _{d_i}),
°F | Outlet
(t _{d_o}),
°F | Average _a
(t _d),
°F | | | | |
| | 10 | | | | | | | | | |
| | 10 | | | | | | | | | |
| | 10 | | | | | | | | | |
| | | | | | | | | | | Y = |

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d

where

V_w = Gas volume passing through the wet test meter, ft³.

V_d = Gas volume passing through the dry gas meter, ft³.

t_w = Temperature of the gas in the wet test meter, °F.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, °F.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, °F.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o} , °F.

ΔH = Pressure differential across orifice, in. H_2O .

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs;
tolerance = pretest Y \pm 0.05Y.

P_b = Barometric pressure, in. Hg.

Θ = Time of calibration run, min.

POSTTEST METER CALIBRATION DATA FORM (Metric units)

Test number _____ Date _____ Meter box number _____ Plant _____

Barometric pressure, P_b = _____ mm Hg Dry gas meter number _____ Pretest Y _____

| Orifice
manometer
setting,
(ΔH),
mm H ₂ O | Gas volume | | Temperature | | | | Time
(Θ),
min | Vacuum
setting,
mm Hg | Y_i | $\frac{V_w P_b (t_d + 273)}{V_d \left(P_b + \frac{\Delta H}{13.6} \right) (t_w + 273)}$ |
|--|---|--|---------------------------------------|-------------------------------|--------------------------------|--|------------------------------|-----------------------------|-------|--|
| | Wet test
meter
(V_w),
m ³ | Dry gas
meter
(V_d),
m ³ | Wet test
meter
(t_w),
°C | Dry gas meter | | | | | | |
| | | | | Inlet
(t_{d_i}),
°C | Outlet
(t_{d_o}),
°C | Average ^a
(t_d),
°C | | | | |
| | 10 | | | | | | | | | |
| | 10 | | | | | | | | | |
| | 10 | | | | | | | | | |
| | | | | | | | | | | Y = |

^a If there is only one thermometer on the dry gas meter, record the temperature under t_d

where

V_w = Gas volume passing through the wet test meter, m³.

V_d = Gas volume passing through the dry gas meter, m³.

t_w = Temperature of the gas in the wet test meter, °C.

t_{d_i} = Temperature of the inlet gas of the dry gas meter, °C.

t_{d_o} = Temperature of the outlet gas of the dry gas meter, °C.

t_d = Average temperature of the gas in the dry gas meter, obtained by the average of t_{d_i} and t_{d_o} , °C.

ΔH = Pressure differential across orifice, mm H₂O.

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs;
tolerance = pretest Y \pm 0.05Y.

P_b = Barometric pressure, mm Hg.

θ = Time of calibration run, min.

METHOD 4 FIELD AND SAMPLE RECOVERY DATA FORM

| | |
|---------------------------|--------------------------------|
| Plant _____ | Probe material _____ |
| Location _____ | Sample box number _____ |
| Operator _____ | Meter box number _____ |
| Date _____ | Meter ΔH @ _____ |
| Run number _____ | Meter cal. (Y) _____ |
| Ambient temperature _____ | Final leak rate _____ |
| Barometric pressure _____ | Vacuum during leak check _____ |
| Probe length m(ft) _____ | Thermometer number _____ |
| | Static pressure _____ |

| | Impinger
volume,
ml | Silica gel
weight,
g |
|---------|---------------------------|----------------------------|
| Final | | |
| Initial | | |

[illegible]

Total

Average

* Acceptable $\Delta V_m = 0.9 < \frac{V_{\text{final}} - V_{\text{initial}}}{\text{number of points}} < 1.1$ _____ to _____.

METHOD 4 ANALYTICAL DATA FORM

Plant _____

Date _____

Run number _____

| | Volume of liquid water collected | | |
|------------------------|----------------------------------|----------------------|----|
| | Impinger volume, ml | Silica gel weight, g | |
| Final | | | |
| Initial | | | |
| Liquid collected | | | |
| Total volume collected | | g* | ml |

* Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml):

$$\frac{\text{Increase, g}}{1 \text{ g/ml}} = \text{water volume, ml.}$$

MOISTURE CONTENT CALCULATION FORM
(English units)

VOLUME OF WATER VAPOR CONDENSED IN IMPINGERS

$$V_f = \text{---} \cdot \text{ml}, V_i = \text{---} \cdot \text{ml}$$

$$V_{wc(std)} = 0.04707 (V_f - V_i) = \text{---} \cdot \text{---} \text{ ft}^3 \quad \text{Equation 6-1}$$

VOLUME OF WATER VAPOR COLLECTED IN SILICA GEL

$$W_f = \text{---} \cdot \text{g}, W_i = \text{---} \cdot \text{g}$$

$$V_{wsg(std)} = 0.04715 (W_f - W_i) = \text{---} \cdot \text{---} \text{ ft}^3 \quad \text{Equation 6-2}$$

SAMPLE VOLUME

$$V_m = \text{---} \cdot \text{---} \text{ ft}^3, T_m = \text{---} \cdot \text{---} ^\circ\text{R}, P_m = \text{---} \cdot \text{---} \text{ in. Hg}$$

$$Y = \text{---} \cdot \text{---}$$

$$V_{m(std)} = 17.64 \frac{V_m Y P_m}{T_m} = \text{---} \cdot \text{---} \text{ ft}^3 \quad \text{Equation 6-3}$$

MOISTURE CONTENT

$$V_{wc(std)} = \text{---} \cdot \text{---} \text{ ft}^3, V_{wsg(std)} = \text{---} \cdot \text{---} \text{ ft}^3,$$

$$V_{m(std)} = \text{---} \cdot \text{---} \text{ ft}^3$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} = \text{---} \cdot \text{---} \quad \text{Equation 6-4}$$

MOISTURE CONTENT CALCULATION FORM
(Metric units)

VOLUME OF WATER VAPOR CONDENSED IN IMPINGERS

$$V_f = \text{---} \cdot \text{ml}, V_i = \text{---} \cdot \text{ml}$$

$$V_{wc(std)} = 0.001333 (V_f - V_i) = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3 \quad \text{Equation 6-1}$$

VOLUME OF WATER VAPOR COLLECTED IN SILICA GEL

$$W_f = \text{---} \cdot \text{g}, W_i = \text{---} \cdot \text{g}$$

$$V_{wsg(std)} = 0.001335 (W_f - W_i) = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3 \quad \text{Equation 6-2}$$

SAMPLE VOLUME

$$V_m = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3, T_m = \text{---} \cdot \text{---} \cdot \text{---} ^\circ\text{K}, P_m = \text{---} \cdot \text{---} \cdot \text{---} \text{ mm Hg}$$

$$Y = \text{---} \cdot \text{---} \cdot \text{---}$$

$$V_{m(std)} = 0.3858 \frac{V_m Y P_m}{T_m} = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3 \quad \text{Equation 6-3}$$

MOISTURE CONTENT

$$V_{wc(std)} = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3, V_{wsg(std)} = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3,$$

$$V_{m(std)} = \text{---} \cdot \text{---} \cdot \text{---} \text{ m}^3$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} = \text{---} \cdot \text{---} \cdot \text{---} \quad \text{Equation 6-4}$$

498

MOISTURE CONTENT CALCULATION FORM
(English and metric units)

MOISTURE CONTENT

t_s avg = _ _ . _ °F, _ _ . _ °C

P_{bar} = _ _ . _ in. Hg, _ _ . _ mm Hg

P_{static} = _ _ . _ in. H_2O , _ _ . _ mm H_2O

S.V.P. = _ . _ in. Hg, _ . _ mm Hg

$$B_{ws} = \frac{S.V.P}{P_{bar} + \frac{P_{static}}{13.6}} = 0 . _ _ _$$

METHOD 4 CHECKLIST TO BE USED BY AUDITORS

| Yes | No | Operation |
|----------|-------|---|
| | | <u>Presampling preparation</u> |
| _____ | _____ | 1. Knowledge of process conditions |
| _____ | _____ | 2. Calibration of pertinent equipment prior to each field test; in particular, the dry gas meter should be checked before each test |
| | | <u>On-site measurements</u> |
| _____ | _____ | 3. Leak testing of sample train after sample run |
| _____ | _____ | 4. Addition of water and silica gel to impingers, and correct location of impingers |
| _____ | _____ | 5. Constant sampling rate and not exceeding specified rate |
| _____ | _____ | 6. Measurement of condensed water to within specified limits |
| _____ | _____ | 7. Record of pertinent process condition during sample collection |
| _____ | _____ | 8. Probe maintained at given temperature |
| | | <u>Postsampling</u> |
| _____ | _____ | 9. Calculation procedure/check |
| _____ | _____ | 10. Calibration checks |
| COMMENTS | | |
| | | |